Vol.
3
1953

VACUUM

A Review of Developments
in
Vacuum Research and Engineering

Vol. III | JULY, 1953 (published in June, 1955) | No. 3

A NEW IMPETUS TO CATHODIC SPUTTERING

THE STUDY OF electrical discharges in gases has yielded a remarkable number of discoveries leading to many practical applications, notwithstanding the fact that research has not yet succeeded in clarifying all aspects of the complex processes occurring in an ionised atmosphere.

Cathodic sputtering is a typical example of a glow discharge applied most extensively and successfully for the deposition of thin metallic films in spite of the fact that the mechanism of sputtering is far from being fully understood. Sputtering is of course normally carried out in the presence of gases at moderate vacuums, and early in the investigations of the technique it was found that sputtered deposits were contaminated in various ways. This was all too readily attributed at the time to defects in the pumping system or lack of cleanliness of the vacuum chamber. Such misinterpretations were probably due to the fact that those engaged in the work were physicists rather than chemists and did not appreciate the chemical reactions which could take place in the glow discharge. However, some early investigators made deliberate attempts to produce chemical compounds by this procedure.

In recent years the very complex nature of sputtering, including the chemical aspects, have been better understood, and it is now generally realised that metal sputtering in an oxygen atmosphere, for instance, invariably forms metal oxide films. The new process, termed *reactive sputtering*, facilitates, among other possibilities, the deposition of transparent conducting films onto glass surfaces—a resistance as low as four ohm per square being readily obtainable with high transparency. Anti-static coatings and electrically heated aircraft screens for de-misting and de-icing purposes are examples of serious problems which are being dealt with by this new method.

The present issue of ' VACUUM ' includes a report on reactive sputtering and the next issue will contain a general survey of sputtering by Professor Güntherschulze, the well-known authority in this field. As a matter of interest, Professor Güntherschulze foreshadowed the method of reactive sputtering when he established at an early stage of his research work that an active discharge gas could react with the cathode to form volatile chemical compounds, *e.g.* hydrides, which dissociated at the receiving surface to give a metal deposit. He called this process " chemical sputtering ".

For some time sputtering has been eclipsed by the evaporating method for the deposition of thin metallic films, but it would seem that the new reactive techniques will open up many new possibilities for the application of the sputtering technique.

LEONARD RIDDIFORD

M.Sc., Ph.D., A.Inst.P.

Research Fellow,
Department of Physics,
University of Birmingham

Dr. Riddiford was born at Port Melbourne, Victoria, Australia, in 1924. He was educated at the Melbourne High School and the University of Melbourne, where he graduated in Physics in 1944 and was granted a Master of Science degree for research in that subject in 1946. After a year as a senior demonstrator in physics he came to England towards the end of 1947 following the award of a scholarship by the Commissioners for the Exhibition of 1851. At Birmingham he was awarded the degree of Doctor of Philosophy in physics in 1950, and has since remained in Birmingham as a University Research Fellow.

His work has been mainly concerned with the design, construction and operation of high energy particle accelerators and experiments with the radiations from them. At Melbourne he took part in the construction of a 3 MeV betatron and an investigation of the gamma radiation from it with a cloud expansion chamber. At Birmingham he was a member of the group responsible for the design, construction and operation of the 1,000 MeV proton synchrotron, his main concern being the responsibility for the vacuum system and the induction electrode for detecting the circulating beam. In the course of this work the theory and practice of oil diffusion pumps and ionisation gauges occupied his attention, and he was able to perform extensive studies on the behaviour of the beam in a proton synchrotron. He also made some general contributions to accelerator design.

More recently Dr. Riddiford has taken part in the work of developing the proton synchrotron to the experimental stage. He has constructed a large high-pressure diffusion cloud chamber and the associated magnet to provide a pulsed magnetic field for it. At present he is engaged in a study of the interactions of the high energy rays from the synchrotron with the protons in the cloud chamber.

the importance of high vacua to HIGH ENERGY NUCLEAR PHYSICS

L. Riddiford, M.Sc., Ph.D., A.Inst.P.,

Physics Department,
*The University, Birmingham.**

Summary

A BRIEF HISTORICAL ACCOUNT is given of the development of cyclic accelerators, and present and future accelerators are discussed. An attempt is made to show the important place which these machines occupy in a most fundamental field of physics. The dependence of both the machines and the physics upon technology, and in particular high vacuum technique, is emphasised.

Sommaire

REVUE HISTORIQUE SUCCINCTE des développements des accélerateurs cycliques et discussion des accélerateurs présents et futurs. Une tentative de demonstration de la place prépondérente occupée par ces machines dans les domaines les plus fondamentaux de la physique est faite. Il est insisté sur la dépendance, et des machines, et de la physique, de la technology et de la téchnique du haut vide en particulier.

Vol. 3 1953

INTRODUCTION

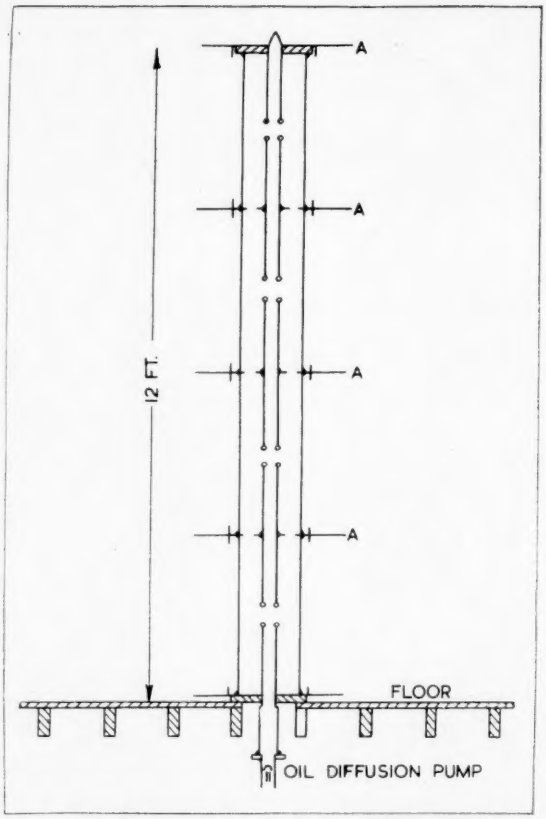
CYCLIC ACCELERATORS at present afford the means of obtaining fundamental particles for use in experiments in high energy nuclear physics with energies more than an order of magnitude greater than those obtainable from non-cyclic accelerators. It seems that in the foreseeable future this difference will widen still further. Apart from the cyclic action, the use of magnetic guiding fields is more efficient than the use of electrostatic ones. The superiority of the cyclic over linear accelerators is, however, obtainable only at the expense of a considerable technical effort in which the emphasis is laid with roughly equal stress upon development in both heavy and precision engineering. To discuss the details of all the techniques affected by this very pronounced trend in modern physics would make unreasonable demands upon both the author of this article and the editor of the journal. It is sufficient to say that the development in the field since it was first entered in 1932 would not have been possible without similar developments in vacuum engineering.

Vacua of the order of 1×10^{-6} mm. Hg have been achieved in very large systems without excessive difficulty, by suitable development of the experience and skill of earlier workers in the field. No doubt still better vacua will be obtainable in the years ahead, but for the moment the tendency is to design the accelerators now taking shape on the basis of present experience and skill.

Although this paper is concerned mainly with cyclic accelerators, it would not be complete without a description of the vacuum tubes of the apparatus[1] with which Cockcroft and Walton[2] first succeeded in causing the artificial transmutation of various elements by high velocity protons. One of the tubes is drawn in Fig. 1, and the description in the original article is as follows :

*MS. received April, 1954.

" Four glass cylinders . . . . . were erected in the form of a tower, and pieces of tinned sheet iron, A, 3 feet square, were placed between adjacent cylinders. The electrodes, B, were attached to these and they also served the purpose of giving a fairly uniform potential gradient down the length of the cylinders. The top and bottom of the tower were closed by thick metal plates. All the joints were made airtight by means of 'plasticine' placed on the outside. When this is worked in tightly with the fingers, quite a good vacuum can be obtained. The surface is then rubbed with tap grease to seal up any minute holes such as might occur along the surface between the glass or metal and the plasticine. This method of making joints has been found to be very reliable. The ease with which the joint may be made and broken again makes it a very convenient type of vacuum joint. The two surfaces to be joined require only a very rough preparation. Thus a good joint between a flat plate and a 3 inch. brass tube can easily be made even if the end of the latter has merely been sawn off roughly with a hacksaw and considerable gaps have to be bridged by the plasticine*. In some preliminary experiments on the large glass cylinders, ordinary commercial plasticine was employed and found to be quite satisfactory. In the final tower of rectifiers, a special putty-like sealing compound was used, which had been made from low-vapour pressure products . . . . . The tube was evacuated by a fast oil diffusion pump, the connection to the pump being made through 3-inch. steam piping."



by courtesy of Sir John Cockroft and Proc. Roy. Soc.

Fig. 1. Cockroft-Walton Vacuum Tube.

In a previous publication[3] *the pump was described as follows:*

" The pump used is a three-stage oil diffusion pump designed by Burch (we are indebted to Metropolitan Vickers Electrical Company for supplying us with several of these pumps before they had been put on the market). The speed of the pump is 20 litres per second and full advantage of this can be taken as no liquid-air trap is necessary. This type of pump has been found to be very satisfactory. Its speed was evident during the outgassing of the anodes. The bombarding potential used caused the tubes to go 'soft' when the gas was liberated. On switching off this potential for only a few seconds, the large volume of gas in the three bulbs was found to have been completely exhausted. The only disadvantage which oil has as compared with mercury is that it is necessary to be careful that air is not admitted to the apparatus while the oil is hot.

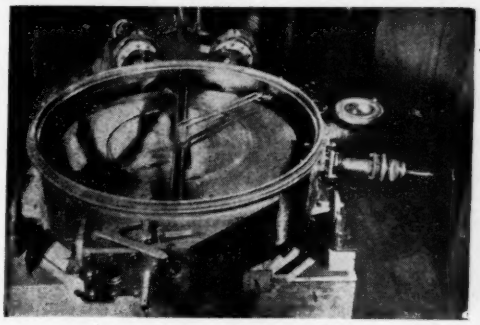
The three bulbs are joined together by waxing them into a steel T-piece. The insulation bulb carries most of the weight and is supported directly on top of the pump. It is connected to the latter by a flat steel ground joint to which it is waxed. . . . . When a puncture through the glass occurs, it is found that the leak produced can be very readily stopped by painting over the hole with cellulose enamel."

* In the experience of the author however, it is necessary that the surfaces on which the seal is made should be quite smooth.

There is little doubt that the designers of current cyclic accelerators would find themselves in considerable difficulty if they followed the vacuum practice described above. However, certain principles still hold. Any apparatus should be designed and made in the simplest possible fashion. In particular, for an experimental piece of equipment the amount of auxiliary apparatus which may cause difficulties at inconvenient times during experiments, should be kept to the minimum consistent with safety. Maximum flexibility of the equipment should be sought within the limit of efficiency imposed by the research funds available.

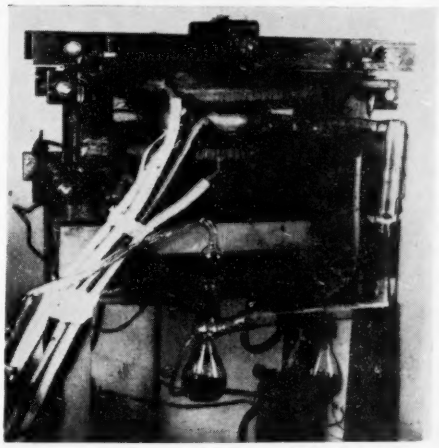
In high tension sets, such as the one used by Cockcroft and Walton or the linear accelerators developed since the war to attain higher energies, the particles are accelerated in straight lines of a length of the order of 10–50 feet. A main purpose of the vacuum in these machines is to avoid the gaseous electrical discharges of either a steady or transient nature which may occur under the intense electric stresses to which the apparatus is subjected. Such discharges limit the voltage obtainable and may damage the equipment. In cyclic accelerators, particularly betatrons and synchrotrons, the particles make many revolutions and the path length may be as great as ten thousand miles. For this reason, vacua about two orders of magnitude better are required to reduce the effects of collisions with the remanent gas atoms.

The degree of vacuum required in a cyclic accelerator to limit the number of collisions depends on several important machine parameters such as injection energy, path length, and the duration of the acceleration period. In cyclotrons and synchrocyclotrons the particles are accelerated very quickly by large voltages and whilst their energy is low they move only short distances on small circles in the strong magnetic field; in betatrons and synchrotrons, the energy gain per revolution is low and the path length is long. But in cyclotrons the effective injection energy corresponds only to the thermal velocity with which the ions are formed, whilst betatrons and synchrotrons use a beam of particles injected at quite a high energy. The result is that the pressure required in the vacuum chamber of a cyclotron or



by courtesy of Revue d'Optique

Fig. 2. Vacuum Chambers of Early Accelerators.
(*a*) Early Berkeley cyclotron.

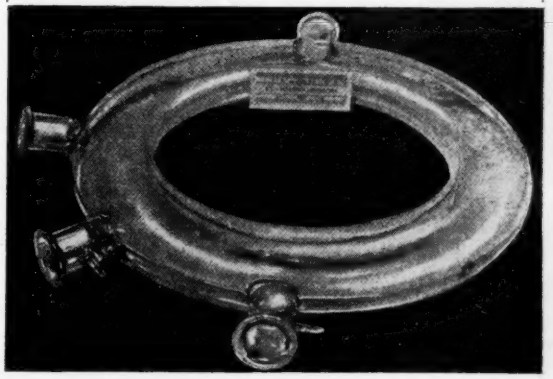


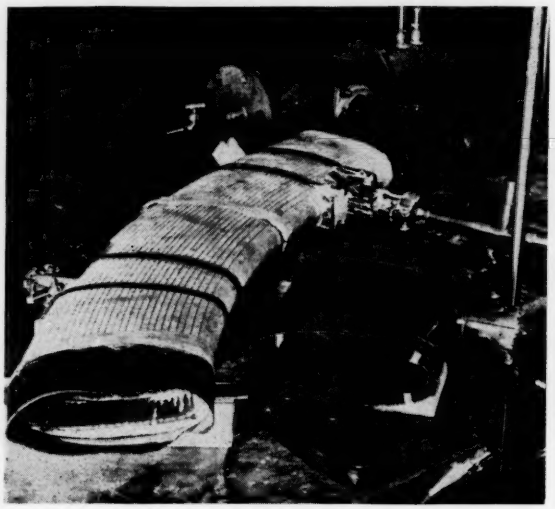
by courtesy of J. Sci. Instrum.

(*b*) Melbourne 3 MeV betatron.

(*c*) 15-inch. betatron doughnut.

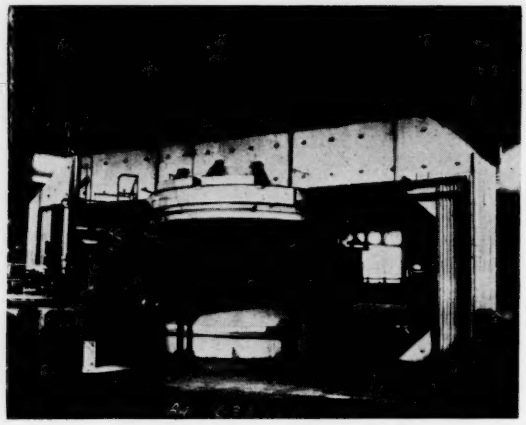
by courtesy of Electronic Engng.





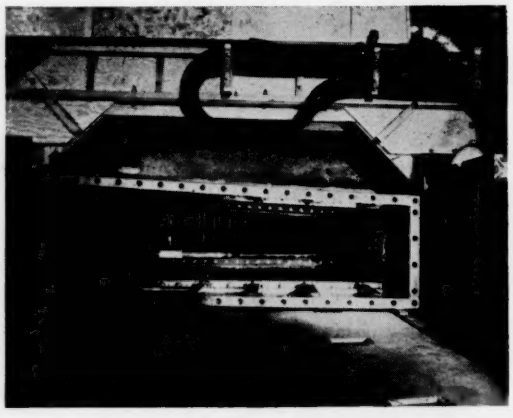
by courtesy of Newman Lab., Cornell University

Fig. 3. Doughnut of a 300 MeV electron synchrotron.



by courtesy of Radiation Lab., University of California

Fig. 5. Vacuum Tank of the 184-inch. synchro-cyclotron.



by courtesy of Radiation Lab., University of California

Fig. 4. (*a*) End View of Vacuum Box of Model Proton Synchrotron.

(*b*) General View of Model Proton Synchrotron.

Vol
3
195

synchro-cyclotron is of the order of 10^{-4}–10^{-5}mm. Hg whilst for a betatron or synchrotron it is of the order of 10^{-5}–10^{-6} mm. Hg.

The earliest cyclotrons used small glass vacuum chambers. Betatrons and electron synchrotrons for energies not greater than about 20 MeV used glass, quartz or ceramic 'doughnuts' fabricated in one piece, and evacuated by small diffusion pumps. Later, cyclotrons used metal chambers, the pieces of which were sealed together with vacuum wax. Fig. 2 shows a few typical units. Photographs of many others can be found in the book by Nahmias[4] on accelerators, including some of the more recent and larger accelerators. The development of the rubber vacuum gasket was an essential part of the systems for larger machines, in which the vacuum tanks and doughnuts were fabricated from many pieces of increasingly complicated design. The Wilson seal is named after a prominent physicist. This seal, by the use of small rubber rings which fit tightly over a shaft passing into the vacuum system, makes possible the movement of the shaft without disturbing the pressure in the system. The added flexibility which results from this facility is essential to the efficient operation of most larger particle accelerators. For a great many applications the Wilson seal has now been superseded by the 'O'-ring seal because of its simpler construction.

Coupled with the increase in size and complication which affect the mechanical engineering aspect of the problem there has always been a demand for better vacua. Early cyclotrons worked at 10^{-4} mm.Hg, the first betatrons operated at 10^{-5} mm. Hg and present proton-synchrotrons operate best at or near 10^{-6} mm. Hg. The importance of gas scattering in cyclic accelerators has been discussed by the present author[5] and others[6]. The most important factors from the vacuum point of view in cyclotrons and synchro-cyclotrons are the need to maintain the dee voltage at the required level by avoiding gaseous discharges, and the loss of beam due to the dissociation of molecular hydrogen ions when these are being accelerated, due to collisions with gas molecules[7]. In betatrons and synchrotrons pure Rutherford-law coulomb gas scattering is the prime consideration.

PRESENT-DAY CYCLIC ACCELERATORS

Electron Synchrotrons

There have been in existence now for several years a number of electron synchrotrons operating in the energy range 300-350 MeV and a betatron for 330 MeV at the University of Illinois represents possibly the most beautiful instrument in the field from the technical point of view. The quarter-scale model proton-synchrotron of the University of California has operated at 500 MeV with electrons at the California Institute of Technology, and will no doubt shortly be running at the full energy of 10^9 eV (1 BeV). In all these instruments a moderate vacuum problem had to be satisfactorily met, the requirements being intimately related to many other aspects of the machines and their design parameters as previously indicated. The doughnut of a typical 300 MeV synchrotron is seen in Fig. 3[8]. The γ-radiation from all these machines is sufficiently energetic to produce π-mesons.

In most of these accelerators a sufficiently high injection energy is obtainable by the use of an electron gun mounted inside the doughnut, to which a pulsed potential of up to 80 kV can be applied. A disadvantage of such an internal gun for very small machines is the heating and outgassing of the walls of the tube if a thoriated tungsten filament is used. The doughnut has to be made of insulating material since the magnetic field varies very rapidly with time and the presence of enough material of sufficient conductivity would result in the creation of eddy currents of some magnitude. These would distort the magnetic field which is designed to vary with radius in such a manner that particles not circulating on the equilibrium orbit will be compelled to move towards it. The vessel has to be sufficiently strong to withstand safely the force of the atmosphere on it when it is evacuated and yet shaped so as to make the best use of the air gap space provided in the magnet, in which particles may circulate. A further requirement is that the inside wall of the doughnut be coated with a thin layer of material (or a thicker laminated layer) sufficiently conducting to ensure the removal of charged particles lost to the wall, the electric field

from which would otherwise destroy the remaining beam. Over a portion of the doughnut these layers form the electrode, to which the accelerating radio-frequency voltage is applied.

The above problems have been solved in a variety of ways, Fig. 3 demonstrating one of them. The doughnut sections are made in quartz, and the irregularities in wall thickness demonstrate the difficulties with which manufacturers are faced. The coating consists of strips of silver fired on the surface, the gaps between strips being coated with a material of lower conductivity. The vacuum seals between sections in this case are made by stretching well-greased broad rubber bands tightly around the outside, the surfaces having to be smooth and convex. The use of the well-known double gasket technique in such a design would seem to be precluded, but the systems are not sufficiently large for this to be very serious.

The vacuum tube of the quarter-scale model synchrooron[9] was constructed from thin corrugated stainless steel sheets strengthened by strong bars insulated from the sheets to minimise eddy currents. In this case the magnetic field increased quite slowly with time. Fig. 4a is an end-photograph of part of the vacuum box, and Fig. 4b shows clearly the extremely high concentration of pumping speed available. A pressure of about 2×10^{-6} mm. Hg was obtained and proved to be only just good enough. The injector was a small, heavy current cyclotron operating at 750 KeV, the beam being well resolved and collimated. At present bursts of electrons are injected into this accelerator from a one-million volt pulse transformer. Apart from the vacuum problem, a very important reason for using a high injection energy with a large machine is that the injection magnetic field would otherwise be small. The inhomogeneities in the magnetic field would then become a more serious cause of trouble than they are at present.

Synchro-Cyclotrons

Following the successful adaptation of the Berkeley 37-inch. cyclotron for operation as an experimental synchro-cyclotron, advance in this field has been rapid. The Berkeley 184-inch. synchro-cyclotron[10] was the first large accelerator of this type and there are now several machines of a similar size operating in the U.S.A., the highest energy being available at Chicago, where a beam of 450 MeV protons is available.* From the Universities of California and Chicago have come very important discoveries and studies about artificially produced π-mesons[11].

These large synchro-cyclotrons normally use, say, three very large diffusion pumps of 20–32 inch. diameter, one of which serves to differentially pump the dee line near the rotating condenser used for modulating the frequency. A detailed account of one of these large machines has been published[12], and Fig. 5 is a photograph which shows the size of the vacuum tank, pumps and valves for the 184-inch. synchro-cyclotron.

The magnetic field in a cyclotron does not vary with time and it is possible to construct the vacuum chamber in one piece by fairly standard techniques. It is usually fabricated from thick stainless steel sheets since steel walls would divert some of the magnetic flux which would otherwise pass through the centre of the vacuum box. This would weaken and distort the main field. In such large systems it is essential to use double gaskets wherever possible so that leak-hunting is simplified and repairs to the system can be deferred to a convenient time by continuously rough pumping the space between gaskets when necessary. To avoid loss of running time, valves for isolating various parts of the system and airlocks for the insertion of probes, targets, etc. into the main chamber are essential. With metal chambers the provision of thin windows through which the various beams of radiation can emerge without loss of energy and partial transformation into other forms of radiation is easier than for an insulating chamber. Similarly minor alterations to the equipment for experimental purposes are easier to achieve.

In a cyclotron or synchro-cyclotron the design of the pumping system must take account of the quite large flow of gas required to supply the ion source. This source is placed (through an air lock) inside and at the centre of the vacuum tank and for this reason

* A 400 MeV synchro-cyclotron is now in operation at the University of Liverpool.

Vol. 3 1953

cannot conveniently be differentially pumped as it can be in the case of proton synchrotrons. The gas is normally hydrogen and because of this the heater power for the difiusion pumps should be specially set at a high value so that the pumping speed is increased substantially above the abnormally low value generally obtained for hydrogen with oil diffusion pumps. For cyclotrons in particular, where the dee voltages are highest and the release of gases and vapours from the dees and the walls of the vessel is severe, it is important to provide the diffusion pumps with one or two powerful booster stages. This is particularly advantageous in reducing the initial pump-down time when the accelerator is being started up after a shutdown.

Another feature essential to a good pump-down time is the provision of adequate baffling. In betatrons and synchrotrons this is of added importance to be sure that the metallised walls of the doughnut remain conducting and do not become unreasonably contaminated with an insulating layer of oil and its decomposition products. Most cyclotrons use refrigerated baffles but in the experience of the author water-cooling of a well-designed baffle should be sufficient. The mechanical pumps should provide adequate pumping speed in the important pressure region from, say, 1.0 to 0.01 mm. Hg.

Care has to be taken to see that the radio-frequency electrodes do not restrict the pumping speed in places inside the main tank where it is important, and for this reason some of the copper conductors carrying the radio-frequency currents may be perforated. One of the modifications shortly to be made to the Berkeley 184–inch. synchro-cyclotron in an attempt to increase the proton energy available to about 710 MeV, will be the insertion of new coils into the vacuum box to provide means for added excitation of the magnet. The gases and vapours released from these coils by the power supplied to them may well take up quite a large part of the available pumping speed.

Proton-Synchrotrons

There are now two proton-synchrotrons in operation, one at Brookhaven[13] for 2.2 BeV protons (shortly to be increased to 2.8 BeV), and one at Birmingham[14] for 1.0 BeV protons. A third accelerator for 6.5 BeV is close to completion at Berkeley*. Already at Brookhaven V-particles† and showers of π-mesons have been artificially produced for the first time.

The vacuum system of all these accelerators is very complicated, as are many other aspects of the machine design. The degree of vacuum required is dependent upon many machine parameters; the Birmingham machine requires about 1.0×10^{-6} mm. Hg but the other two can tolerate pressures near 1×10^{-5} mm. Hg. Fig. 6 shows results of preliminary studies of the Birmingham synchrotron's performance, at various pressures and with the doughnut aperture limited at various values by wire stops inserted through an air-lock. The outstanding feature is that the machine ceases to function at an air pressure of 2.5×10^{-6} mm. Hg, and for good operation the average pressure in the doughnut must not exceed about 1.3×10^{-6} mm. Hg. This result is in very good agreement with the calculations of Blachman and Courant[16], when allowance is made for the fact that the magnetic median plane and thus the beam of protons is substantially below the geometric median plane of the magnet, and also that there is a substantial forced instability in the phase oscillations near the beginning of the acceleration cycle. A preliminary account of the requirements and design of this vacuum system was published in 1951[17] and the construction and operation will be described elsewhere. Fig. 7 is a photograph of the accelerator with the main features of the vacuum system apparent. It is sufficient to say that except for short periods, without the aid of refrigeration of any kind, the pressure has been maintained at about 1.0×10^{-6} mm. Hg. for eighteen months without undue difficulty.

There has been no trouble of the kind which would arise from any accumulation of charges on the platinised walls of the porcelain doughnut. The use of double vacuum gaskets has been justified and a procedure such as that previously described for a 300 MeV electron synchrotron would have proved

* This machine is now operating.

† The new particles have recently been re-classified[15] by a group of leading cosmic-ray physicists.

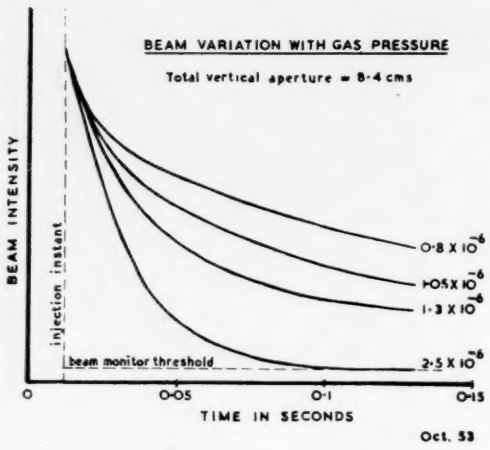


Fig. 6. (*a*) Effect of varying pressure.

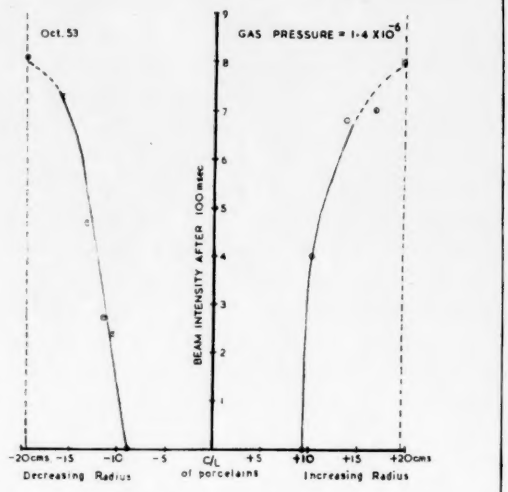


Fig. 6. (*c*) Effect of varying horizontal aperture.

Results of preliminary beam studies in the Birmingham proton synchrotron.

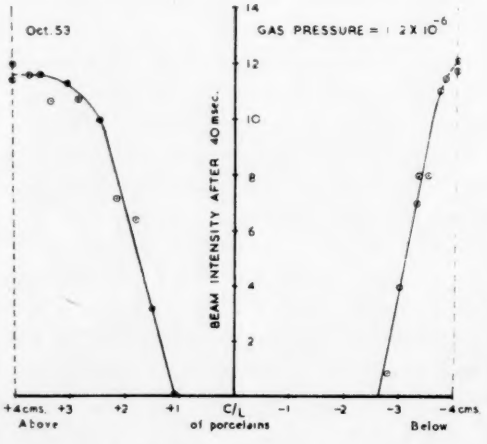


Fig. 6. (*b*) Effect of varying vertical aperture.

fatal. Immediately following assembly the seals between the sixty porcelain sections from which the doughnut is fabricated proved to be quite sound. After some weeks however, it was found necessary to pump out the double gaskets continuously to maintain the very low leak rate of the order of one micron-litre per second required. Presumably the thin film of grease with which the gaskets were smeared has been absorbed by the rubber. No one gasket is troublesome but the combined effect would be fatal if not attended to. The leak would not be sufficiently large to attract attention in most cases and causes no embarrassment to us, for the pump-out system is an elementary one and quite reliable.

The vacuum system of the Brookhaven synchrotron has also been described[13]. This differs considerably from the Birmingham one in design and neither the pressure required nor obtained has been less than 5×10^{-6} mm. Hg. The limit is set mainly by the vapour pressure and/or the porosity of the (special) rubber blankets which form the vacuum-tight cloak for the tube. These are stretched over the rigid laminated stainless steel structure which provides the mechanical strength to withstand the atmospheric

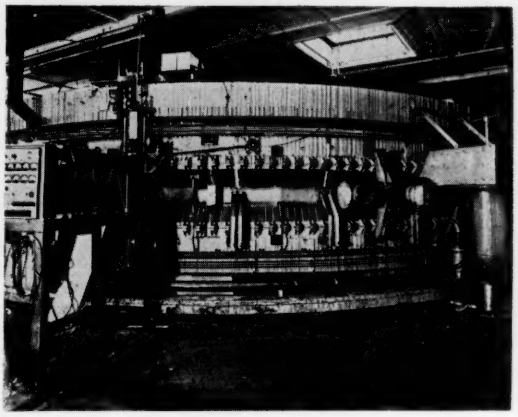
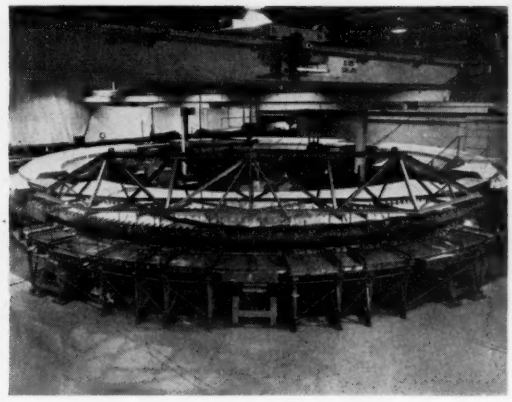


Fig. 7. The Birmingham Proton Synchrotron.



by courtesy of D. D. Jacobus, I. J. Polk, & Rev. Sci. Instrum.

Fig. 8. The Brookhaven Cosmotron.

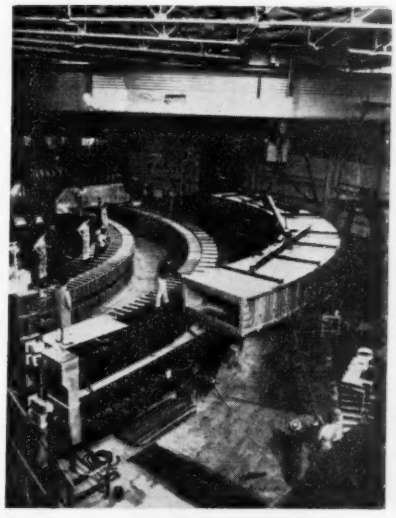
Vol.
3
1953

load when the tube is evacuated. The components of this structure are insulated one from the other to restrict eddy currents. The tube is constructed in four quadrants and each cloak is a continuous sheet of rubber extending for the length of a quadrant. The system uses twelve 20-inch. diffusion pumps compared to five 16-inch. pumps at Birmingham. Fig. 8 is a photograph of the accelerator.

The magnet is in the form of a race track, with four quadrants of a circle, separated but connected by straight sections, rather than a circle as at Birmingham. This results in a longer path length per revolution without added acceleration, and in the straight sections there is no focussing action by a magnetic field. Consequently, the degree of vacuum required is rather higher than would be the case for the corresponding circular machine. The straight sections however, provide a much freer means of access into the vacuum tube than is available at Birmingham, and should make possible additions and alterations to the system which will be of value to the experimental programme.

At Berkeley the system, of necessity again, differs greatly in detail from the Birmingham design

Typical design features of three well-known cyclic accelerators.



by courtesy of Radiation Lab., University of California

Fig. 9. The Berkeley Bevatron.

although not so greatly from the Brookhaven one. Fig. 9 is a photograph of the first quadrant of the vacuum box being moved into place into the H-shaped magnet. The magnitude of the effort in vacuum engineering involved is evident from this photograph. This is due in part to the size of the accelerator and in part to the fact that the pole pieces of the magnet structure are to be assembled inside the

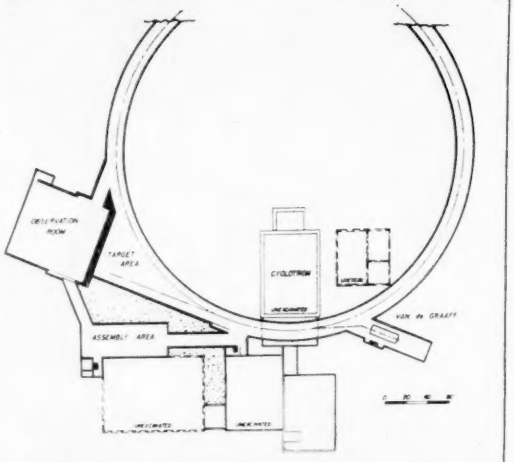


Fig. 10. (*a*), (*b*), (*c*) *by courtesy of Mass. Inst. of Technology*

Fig. 10. (*a*) Plan for a 15 BeV strong-focussing proton synchrotron.

box. They are part of it in that they are used as stiff beams to carry the very great load which the atmosphere will exert upon the vessel. Since the walls of the vessel are placed between the magnet yoke and the pole pieces, the effect of the eddy currents in them upon the field in the magnet gap is less than would otherwise be the case. For this reason a thicker stainless steel sheet than would have been possible at Birmingham or Brookhaven may be used. An early design figure was that thirty-two 32-inch. diffusion pumps were to be employed and if this is still true, the pumping speed available is of the order of 800,000 l./sec. Of course not all of this will be available as effective speed in the tube.

The magnet of the H-shaped cross section is to be compared with the C-shaped magnets in use for the other two accelerators. Unless the top of the magnet yoke is removed the vacuum box is quite inaccessible except in the straight sections, and it is here that the pumps are placed. Such a procedure would have resulted in a substantial loss of pumping speed at the centres of the quadrants in the Brookhaven case, but at Berkeley no space is taken up by a vacuum tube between the poles of the magnet, so that the effect is not as great. The reduction in accessibility is compensated for by the higher efficiency of the H-type magnet, and in any event, for such a very large accelerator the straight sections should provide adequate experimental facilities.

CYCLIC ACCELERATORS OF THE FUTURE

Proton-Accelerators and the Strong Focussing Principle[18]

The method of constructing accelerators for energies of 10 BeV or more became clear in 1953 when Livingston and others presented a design outline for a 30 BeV strong focussing proton-synchrotron[19]. By constructing the guiding magnet in a novel manner it should be possible to confine the particles much more closely to the stable orbit than in previous accelerators, and thus obtain for a similar outlay in cost and physical effort almost an order of magnitude increase in energy. The new (or rather greater amount of) technical development and effort necessary is formidable, but in the light of the operating experience now available from conventional proton-synchrotrons, it is hoped that it will be no more formidable than were the problems faced by these conventional machines themselves in the beginning.

In Fig. 10 are drawn the general plan of a proposed 15 BeV accelerator and cross sections of the magnet and vacuum tube. These were taken from a detailed design study published by a group at the Massachusetts Institute of Technology[20]. A similar detailed design for a 20–30 BeV accelerator is in progress at Geneva under the auspices of the CERN[18,21]. Machines with radii of the order of 150 yards will be constructed with doughnuts of a cross section of about

Vol 3 195

4×2 inch. The main difficulty with these accelerators is likely to be the alignment of the magnet to a sufficient degree of accuracy, for errors in the construction may cause much more trouble than in conventional machines. A further feature is that the protons have to be injected more accurately on to the equilibrium orbit than they do on to the outside orbit in the conventional case. Using a 4 MeV injector for the 15 BeV machine would require a vacuum of about 1.5×10^{-6} mm. Hg. For the 30 BeV machine it is proposed to inject from a proton linear accelerator at a substantially higher energy to overcome the magnet difficulties, and in this case the vacuum problem would be a straightforward one. Since the doughnut is very extended, the order of 500 pumping ports of the order of 3 inch. diameter will be required. A gas scattering theory for the new accelerators has been worked out by Courant[20] and no doubt will prove to be as close to experience as the theory for the conventional machines. It is of interest to note that the injection energy that would be required for a conventional synchrotron of dimensions identical to those of the 15 BeV accelerator mentioned above would be about 100 MeV, were the pressure in the doughnut the same in each case.

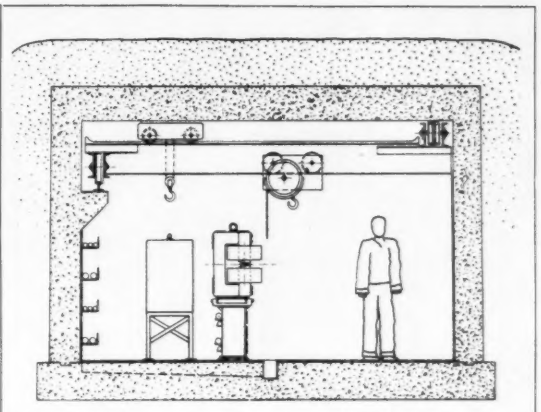


Fig. 10. (*b*) Cross section of 15 Bev accelerator magnet.

Other Accelerators

Most designers of cyclic accelerators bear constantly in their minds the knowledge that a substantial saving in building space is possible with machines which use guiding fields produced from air-cored rather than steel magnets. This is because in the extreme relativistic region the energy of a particle is directly proportional to the product of the radius of its orbit and the strength of the magnetic field in which it moves. Iron is used to reduce both the number of ampere-turns required to excite the magnet coils and the energy stored in the magnetic field. By increasing the field substantially above the value at which the iron effects a worthwhile economy, the radius of the magnet can then be reduced substantially for the same particle energy. The iron is then best dispensed with. Unfortunately the saving in space is more than counter-balanced by the increase in power and stored energy required to excite the magnet. Workers at the G.E.C. laboratories in Schenectady have operated a 300 MeV air-cored electron synchrotron, and Oliphant and others at the Australian National University are studying in detail the design of a 10–15 BeV proton-synchrotron with an air-cored magnet. The field would be of the order of 100,000 gauss on a diameter of the order of 30 feet.

The vacuum system of the G.E.C. synchrotron uses a metal tank in which the air-cored coils are

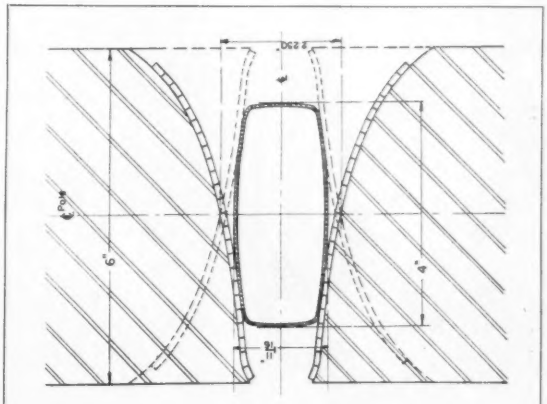


Fig. 10. (*c*), Cross section of 15 Bev accelerator doughnut.

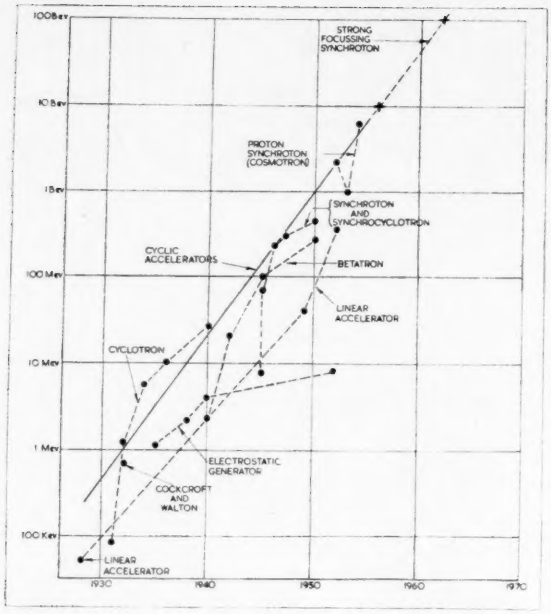


Fig. 11. Chronological plot of the particle energy available from accelerators.

placed. A very large pumping speed into the tank is available and necessary because of the presence of the coils and the supports necessary to withstand the magnetic forces on them. This of course does not guarantee sufficient pumping speed into the accelerating space, which is within the confines of the coil itself. It is possible that the tank for a larger accelerator would be of a similar nature and that the vacuum required would be of the order of 1×10^{-6} mm. Hg.

Workers at Cornell University have completed the construction of a 1.5 BeV electron-synchrotron which uses a very small steel magnet and vacuum tube[22], and a strong focussing electron-synchrotron for 1 BeV of a rather less bold design is also under consideration at Stockholm. The limit to the energy obtainable from electron accelerators will probably be set by the fact that electrons with extreme relativistic energies radiate large quantities of energy when compelled to move on confined orbits in a magnetic field. For a 1.3 BeV accelerator this energy amounts to about 100 KeV per revolution of the particle, and unfortunately increases as the fourth power of the particle energy. The radiated energy must be of course supplied in the form of extra accelerating potential, and the power losses incurred in supplying the potential soon become very large.

Concluding Remarks

Each increase in the energy of the particles available to bombard atomic nuclei has led to the discovery of important facts of great value to nuclear physics. There is no reason to believe that this will not continue to be so in the future. The main difficulty which the future presents at the moment is the fact that what is important in high energy physics is not the particle energy in the laboratory system of co-ordinates (experiments are of course done with a target at rest in this system), but the energy in the centre-of-mass system of the two particles taking part in the reaction in question. For extreme relativistic particles colliding with stationary particles of the same mass, it may be shown that

$$E_{cM}=(2\ M_o c^2\ E_{Lab})^{\frac{1}{2}}$$

so that a proton of energy 100 BeV in the laboratory system provides an energy of only 13 BeV in the centre-of-mass system.

Blewett has plotted the energy available to experimenters from typical accelerators, as a function of time and an extension of the curve is shown in Fig. 11. The important feature of the data is that the curve for cyclic accelerators lies above those for linear accelerators and that it is approximately a straight line on the log-log plot. It is not unreasonable to expect a laboratory source of 100 BeV protons by about 1962. However for still higher energies, approaching that of the fastest cosmic ray particles, a completely new process may well be necessary.

CONCLUSIONS

The past, present and future development of particle accelerators of the cyclic type has been reviewed in the light of existing knowledge. The art has been and will continue to be intimately associated with developments in many branches of technology,

Vol
3
195

of which the science of vacuum engineering is one of the most important. In many instances substantial advances in this technology have come directly from the needs of the accelerator designer and this will continue to be so.

Obvious corollaries are that high energy nuclear physics has relied very much upon technological developments, and that the needs of fundamental physics have had a very great influence upon both the technology of particle accelerators and all subsidiary techniques.

References

1 COCKROFT, J. D. and WALTON, E. T. S., *Proc. Roy. Soc.*, **136A,** (1932), 619-630.
' Production of High-Velocity Positive Ions '.

2 COCKROFT, J. D. and WALTON, E. T. S., *Proc. Roy. Soc.*, **137A,** (1932), 229-242.
' Experiments with High-Velocity Positive Ions. Part 2. Disintegration of Elements by High-Velocity Protons '.

3 COCKROFT, J. D. and WALTON, E. T. S., *Proc. Roy. Soc.*, **129A,** (1930), 477-489.
' Experiments with High-Velocity Positive Ions '.

4 NAHMIAS, M. E., *Les Machines Atomiques*, (Editions de la Revue d'Optique, Paris, 1950).

5 RIDDIFORD, L., *Proc. Phys. Soc.*, **64A,** (1951), 10-12.
' The Importance of Gas Scattering in Particle Accelerators '.

6 FREMLIN, J. H. and GOODEN, J. S., *Rep. Progr. Phys.*, **13,** (1950), 295-349.
' Cyclic Accelerators '.

7 EFFAT, K. E. A., *Proc. Phys. Soc.*, **65A,** (1952), 433-436.
' Dissociation of Molecular Hydrogen Ions in the Cyclotron '.

8 CORSON, D. R., DEWIRE, A. W., MCDANIEL, B. D. and WILSON, R. R., *Floyd Newman Lab. of Nuclear Res. Rep.*, Cornell University, (1953).
' The Cornell 300-MeV Synchrotron '.

9 SEWELL, D. C., LAWRENCE, E. O. and LOFGREN, D. J., *Phys. Rev.*, **78,** (1950), 85.
' Design of the Bevatron Quarter Scale Operating Model '.

10 BROBECK, W. M., LAWRENCE, E. O. and MACKENZIE, *Phys. Rev.*, **71,** (1947), 449-450.
' Initial Performance of the 184-inch. Cyclotron of the University of California '.

11 POWELL, C. F., *Rep. Progr. Phys.*, **13,** (1950), 350-425.
' Mesons '.

12 ANDERSON, H. L., MARSHALL, J., KORNBLITH, L., Jr., SCHWARCZ, L. and MILLER, R., *Rev. Sci. Instrum.*, **23,** (1952), 707-728.
' Synchrocyclotron for 450-MeV Protons '.

13 BLEWETT, M. H., *Rev. Sci. Instrum.*, **24,** (1953), 725-737.
' The Cosmotron—A Review '.

14 ANON., *Nature*, **172,** (1953), 704-706.
' Proton Synchrotron of the University of Birmingham '.

15 AMALDI, E., ANDERSON, C. D., BLACKETT, P. M. S. FRETTER, W. B., LEPRINCE-RINGUET, L., PETERS, B., POWELL, C. F., ROCHESTER, G. D., ROSSI, B. and THOMPSON, R. W., *Nature*, **173,** (1954), 123.
' Symbols for Fundamental Particles '.

16 BLACHMAN, N. M. and COURANT, E. D., *Phys. Rev.*, **74,** (1948), 140-144.
' Scattering of Particles by the Gas in a Synchrotron '.

17 RIDDIFORD, L., *J. Sci. Instrum.*, (Suppl. I, 1951), 47-57.
' Vacuum Physics. The Vacuum System of the Birmingham Proton Synchrotron '.

18 EUROPEAN COUNCIL FOR NUCLEAR RESEARCH, (Proton Synchrotron Group). Conference at the University of Geneva (1953), *U.S.A.E.C. Rep.* No. NP-5070.
' Lectures on the Theory and Design of an Alternating Gradient Proton Synchrotron '.

19 COURANT, E. D., LIVINGSTON, M. S. and SNYDER, H. S., *Phys. Rev.*, **88,** (1952), 1190-1196.
' The Strong-Focussing Synchrotron, a New High Energy Accelerator '.

20 LIVINGSTON, M. A., *et al.*, *Massachusetts Institute of Technology Tech. Rep.* No. 60, (1953).
' Design Study for a 15-MeV Accelerator '.

21 FRY, D. W., *Nature*, **172,** (1953), 646-647.
' An International Laboratory for Nuclear Research '.

22 BLEWETT, M. H., *Brookhaven National Lab. Rep.* No. BNL-213, (1953).
' Notes on the High Energy Accelerator Conference, December, 1952 '.

Vacuum, Vol. III No. 3 *July, 1953*

REACTIVE SPUTTERING
and associated plant design

L. Holland &
G. Siddall.

Research Laboratories, Edwards High Vacuum Ltd.
*Crawley, Sussex**

Summary

THE TECHNIQUE, termed *reactive sputtering*, of preparing thin films of metallic compounds, notably metal oxides, by sputtering from metallic cathodes in the presence of active gases is described and compared with other deposition processes. The oxidation mechanisms which may occur during the sputtering of a metal in an atmosphere containing oxygen are considered. The desirable performance features of a reactive sputtering plant are enumerated. Experience of producing a range of metal oxide coatings has shown that in order to obtain films of a specified chemical composition the concentration of the desorbed gases in the sputtering atmosphere must be reduced to a minimum. This can be achieved by the use of a vapour pump, having a sufficiently high speed at the sputtering pressure to permit a high rate of leakage of the desired gas into the sputtering chamber. The composition of films of metal oxides which do not obey the law of fixed proportions can be varied by sputtering in oxygen plus inert gas mixtures of different concentrations. A simple method for controlling the composition of such gas mixtures is described. Consistent control of the sputtering rate requires the provision of a stable glow discharge operating at a given applied voltage and current density. It is shown that a booster vapour pump used in conjunction with a high vacuum isolation valve of variable aperture permits fine control of the sputtering pressure on which the discharge voltage and current are dependent, also, that a stable glow discharge can be easily obtained if the high tension transformer is of the high reactance type.

Sommaire

LA TECHNIQUE DENOMMEE *projection cathodique réactive* pour la préparation de composés métalliques, notamment d'oxydes, au départ d'une cathode métallique, en présence de gaz actifs, est décrite et comparée à d'autres procédés de dépôt. Les mécanismes d'oxydation pouvant se produire pendant la projection cathodique d'un métal en atmosphère contenant de l'oxygène sont considérés. Les genres de performances exigées d'un appareil à projection cathodique réactive sont énumérés. L'expérience dans la production d'une gamme de couchse d'oxyde métalliques a démontré, que, pour l'obtention d'un film d'une composition chimique donnée, la concentration des gaz désorbés dans l'atmosphère de décharge doit être réduite à un minimum. Ceci peut être réalisé par l'emploi d'une pompe à diffusion ayant une vitesse suffisament élevée à la pression de projection cathodique pour permettre un degré élevé d'infiltration du gaz désiré dans la chambre à décharge. La composition de films d'oxydes métalliques qui n'obéissent pas la loi des proportions fixes peut être variée par décharge en atmosphère d'oxygène additionnée de mélanges de gaz inertes sous différentes concentrations. Une méthode simple de réglage de la composition de tels mélanges gazeux est décrite. Le contrôle continue du degré de projection demande la mise en jeu d'une décharge ionisante fonctionant sous une tension et une densité de courant donné. Il est démontré que l'emploi d'une trompe à vapeur d'huile combinée à une vanne à haut vide d'ouverture réglable permet un réglage fin de la pression de projection cathodique dont dépendent tension et courant. Il est également démontré qu'une décharge ionisante stable est facilement réalisable si le transformateur haute tension est du type à forte réactance.

INTRODUCTION

IT WAS FOUND by many early investigators of the cathodic sputtering technique that thin films deposited from metal cathodes often had anomalous physical properties. Sputtered films of the base metals in particular usually showed optical interference colours and either lacked or had a low electrical conductivity. Few of these workers appreciated that the deposits they were examining were not pure metallic films but rather metallic compounds formed by a chemical reaction between the sputtered metal and active gases in the glow discharge atmosphere. Thus when a glow discharge is operated in a residual atmosphere of air, the sputtered deposits of the more chemically active metals are either oxidised metal or mixtures of oxides and nitrides. The possibility of producing oxide

* MS. Received February, 1955.

films by sputtering in oxygen was foreseen by Overbeck[1] in 1933, but it is only recently that the otherwise troublesome nature of oxide contamination has been turned to advantage for the preparation of metal oxide films. This method of preparing metal oxide films has been termed by Veszi[2] *reactive sputtering*. There is, as already mentioned, the possibility that metal compounds other than oxides may be formed if gases other than oxygen are present in the residual atmosphere. For example, hydrides and sulphides have been reported by workers who have studied electro-chemical reactions in gaseous discharges, see Glockler and Lind.[3] Thus the descriptive name 'reactive sputtering' embraces a much wider field than that of oxide formation alone. However, the first exploitation of the technique has been for the production of metal oxide films.

Vol. 3 1953

ADVANTAGES OF REACTIVE SPUTTERING

Various techniques have been evolved for the preparation of metal oxide films in an attempt to extend the range of dielectric materials which can be deposited in thin film form. A number of inorganic metallic compounds, such as fluorides, sulphides and chlorides, can be vacuum evaporated and these materials are used in optical interferometry for multilayer filters and anti-reflection coatings. For some purposes, such as the construction of beam splitters, the materials which can be vacuum evaporated to form non-absorbing layers of high refractive index, *e.g.*, zinc sulphide or lead chloride, invariably possess poor mar resistance and are attacked in humid atmospheres. Some metal oxides, *e.g.* those of titanium, bismuth and tin etc., are free from these defects, and further have the advantage of being highly adherent to glass surfaces, but they cannot be easily deposited by vacuum evaporation.

The vacuum evaporation of a metal oxide in its highest state of oxidation is often prevented by chemical reactions occurring at high temperatures between the metal oxide and the heater material, which invariably leads to the reduction of the evaporant to a lower oxide or free metal. Also, for some oxides, the oxygen pressure during evaporation may be lower than that required for their stable existence. For example aluminium oxide (Al_2O_3) is a highly stable compound with a dissociation pressure as low as 1.5×10^{-18} mm. Hg at an evaporation temperature of about 2,000°C, but this oxide is partially reduced when it is raised to the evaporation temperature in contact with a tungsten filament heater. On the other hand, titanium dioxide decomposes and forms a lower black oxide when heated *in vacuo* without making a high temperature junction with a refractory support and cadmium oxide decomposes in the atmosphere when heated to 700°C. Some of the methods used for preparing metal oxide films in the highest state of oxidation together with details of their limitations are summarised below :—

(1) *Evaporating from a platinum heater* to prevent decomposition of an oxide due to reaction with the support. The oxides of the alkaline earths can be evaporated substantially in oxide form from platinum heaters, see Blewett[6] and Pelcowitch[7]. It is also claimed that the tungsten oxide WO_3 will evaporate without reduction from a platinum source. The technique is of restricted value because the evaporation temperature of most metal oxides is near to, or exceeds, the melting point of platinum.

(2) *Slowly evaporating a volatile lower oxide* so that the condensed film is free to absorb oxygen from the residual atmosphere during its growth. This technique is currently used for preparing silicon dioxide and titanium dioxide films by evaporating the lower oxides in each case. There is evidence, however, that films prepared in this manner never completely attain the highest state of oxidation.

(3) *Heat oxidising a metallic film in air.* This technique has proved useful for the production of high reflecting and durable titanium dioxide beam splitters, see Hass[8] and Holland[9] *et al.* and iron oxide beam splitters, see Hammer[10] and Holland *et al.*[11]. For optical purposes this method can only be used where, (i) it is permissible to raise the glass support to a high temperature, generally in the region of 300–400°C, and (ii) the structure of the heat

oxidised film does not become granular during its formation, *e.g.*, when zinc cadmium and tin coating are heat oxidised they tend to form granular oxide layers which scatter incident light.

(4) *Depositing an oxide film from a vapour cloud* produced by a chemical reaction between a volatile metal halide and water vapour. This process is currently used for preparing transparent conducting tin oxide films on glass (see reference[12,13,14]). Control of the deposition rate is somewhat of an art and the process must be carried out in a fume cupboard to remove the reaction products.

Reactive sputtering has several advantages over the foregoing techniques. After limited experiment it is usually possible to prepare optically non-absorbing metal oxide deposits to specified film thicknesses. Films of metal oxides which either cannot be evaporated or prepared by heat oxidation of metal films, as described previously, can be sputtered to form coatings of smooth structure. Whilst it may be desirable to raise the temperature of a substrate to make the deposit assume a given crystalline structure, it is usually not a requirement of the oxidation process as in the production of heat oxidised films. Unfortunately metal oxides possessing a low refractive index of approximately n=1.3 are not available for interferometry, but sputtered oxide films (approximately n=2) could be used in combination with evaporated low index materials, *e.g.* magnesium fluoride, to provide durable multi-layer filters.

Films of most of the metal oxides can be deposited at rates which are comparable to the sputtering rates of pure metal films. Thus, using an applied voltage of 3 kV and a current density of 5 mA/cm^2, quarter wavelength films of bismuth oxide and cadmium oxide can be deposited in some 2 to 3 minutes. Such rapid deposition of metal oxides is comparable to the rate which is possible by vacuum evaporation. Aluminium oxide and titanium dioxide require abnormally high current densities of the order of 20 mA/cm.^{2} to obtain these high sputtering rates; the reactive sputtering of aluminium oxide has been discussed by Koenig and Heisinger[5].

The range of metal oxides which have been produced by the reactive sputtering technique is extensive, *e.g.*, Perrot[14] has deposited chromium trioxide. Koenig and Heisinger[5] have studied the physical properties and crystalline structure of oxides of aluminium, thorium, silicon, cadmium, titanium, iron and also mention the deposition of oxides of magnesium, tantalum, beryllium and cerium. Hammer[10] has also prepared a range of oxide layers by reactive sputtering.

Mr. J. S. Preston* in a private communication to the writers has stated that oxides of the following metals have been reactively sputtered without difficulty in his laboratory—cadmium, lead, tin, zinc, copper, tungsten, thallium, indium, tellurium, bismuth, antimony and molybdenum. Of these the oxides of tin, zinc, tungsten, thallium, indium, tellurium, bismuth and antimony are practically transparent, *i.e.* they have an absorption of about 1 or 2% at most for white light with between a $\lambda/4$ to $\lambda/2$ in optical thickness, whereas the sputtered oxides of cadmium and lead are a little more absorbing especially for blue light.

We have sputtered oxides of titanium, platinum, iron, indium, bismuth, tin, nickel and cadmium and the experience gained during this work enables us to deal in a later section of this paper with the design of reactive sputtering plant for general deposition purposes. A report on the physical properties and deposition characteristics of specific oxide films is in preparation and will be published later.

GAS—METAL REACTIONS OCCURRING DURING SPUTTERING

Several conflicting theories have been put forward to explain the *physical* process of cathodic sputtering and although the cause and effect are understood, there is no generally acceptable theory of the mechanism by which metal atoms are released from a cathode when exposed to positive ion bombardment. The problem becomes even more complicated when chemical reactions in the glow discharge have also to be considered. Most reactive sputtering investiga-

* National Physical Laboratory, Teddington.

tions have been concerned with the properties and applications of sputtered metal oxide films rather than with the physical and chemical processes occurring during their formation and the views expressed on the oxidation mechanism in this section cannot be taken as fully established.

When sputtering from a metal cathode in an oxygen atmosphere, there are three possible mechanisms by which a metal oxide film can be formed, all three of which may occur simultaneously :—

(i) The initial formation of an oxide layer on the cathode surface by bombardment with oxygen molecules or ions, which is removed in molecular form during subsequent bombardment.

(ii) Oxygen absorption by sputtered metal atoms during transit to the receiver due to collisions between gas molecules and metal atoms.

(iii) Absorption of oxygen molecules impinging on the deposited film during its condensation.

These processes are accelerated by the presence of ionised gas, which is usually much more active.

Oxidation of the surface of the cathode in an oxygen discharge may be caused either by direct absorption of gas, or by metal oxide molecules formed by collisions between sputtered metal atoms and active gas atoms diffusing back to the cathode. An oxide layer on the surface of a metal cathode often becomes visible as an interference film or as an optically absorbing coating after the cathode has been bombarded in oxygen and when this oxidised or 'activated' cathode is bombarded in an inert gas, *e.g.* argon, an oxide film is deposited until the cathode oxide coating has been removed. Thus if the oxide layer on the cathode is sufficiently thick a metal oxide film can be deposited without the need for oxygen in the residual atmosphere; Preston[15] has prepared cadmium oxide-films using this method. It is therefore reasonable to suppose that an oxide film may be directly sputtered from an oxide coated cathode and in fact this phenomenon has been observed by Takeya *et al.*[16] in gas filled thermionic valves. It also follows *that to produce pure metal films by sputtering it is not sufficient to use an inert gas atmosphere but that the cathode must be bombarded to remove oxide layers before commencing metal deposition.*

It can also be shown that a delay or activation period exists before a fully oxidised metal film is deposited in an oxygen atmosphere. For example, if the oxide layer has been removed completely from a cathode surface by bombardment in argon and oxygen is introduced afterwards into the coating chamber, then the sputtered deposits show a gradual increase in their degree of oxidation as sputtering proceeds.

Whether a stable oxide film can form on the surface of a cathode depends upon its temperature, the nature of the metal and the oxygen pressure. Under certain circumstances, an oxide film cannot exist on the cathode surface, but an oxide film may be deposited by sputtering from such a cathode. For example, when silver is sputtered in an oxygen discharge the cathode surface is unlikely to be oxidised if its temperature rises appreciably because of the thermal instability of silver oxide. However, silver oxide may be formed on the relatively cooler receiving surface due to active oxygen atoms impinging on the growing film. Also, the noble metal platinum readily forms a brown surface layer of oxide on the cathode which sputters and contaminates the metallic deposit when the glow discharge atmosphere contains oxygen.

When the receiving surface is bombarded with gas molecules the metal film may be oxidised or oxygen molecules may be trapped in the metal or oxide lattice during the formation of the film. It is of importance to note that most metal oxides can exist with an excess or deficit of one component, *i.e.*, the composition of the oxide is not strictly stoichiometric. When oxide films are prepared by reactive sputtering the state of oxidation can be controlled by sputtering in a mixture of oxygen with an inert gas and varying the concentration of the oxygen component. For example Helwig[17] found that the state of oxidation of sputtered CdO–films depended upon the concentration of oxygen in the residual atmosphere.

Inert-Active Gas Mixtures

Mixing oxygen with an inert gas such as argon to form the discharge atmosphere has two effects. Firstly, the sputtering rate is usually increased and secondly, the degree of oxidation of the deposited film

Vol. 3 1953

decreases as the partial pressure of oxygen in the gas mixture falls. In applications where an excess of metal is required in the oxide lattice to enhance the conductivity this can be obtained by carefully controlling the proportion of active gas in the mixture. Thus when sputtering from cadmium cathodes, poorly conducting highly oxidised cadmium oxide films are deposited in pure oxygen, whereas highly conducting cadmium oxide films containing an excess of metal atoms are deposited in an argon atmosphere containing a small fraction of oxygen.

If the inert gas in a gas mixture is replaced by hydrogen then any metal oxide formed may be reduced by active hydrogen in the discharge. Some oxides such as aluminium oxide are not reduced by active hydrogen. Koenig and Heisinger[5] have found that it is immaterial whether aluminium oxide is formed by sputtering in a mixture of oxygen and argon or oxygen and hydrogen. It is to be expected that the oxide sputtering rate would be greater with the argon mixture, because it has been generally found in the sputtering of metal layers that the disintegration rate increases with the molecular weight of the gas.

Effects of Degassing from the Apparatus

The sputtering atmosphere will be contaminated by gases and vapours liberated from the chamber walls and this may affect the composition of the deposited oxide film. Some metals readily form nitrides and these may be present in the oxide deposit unless the chamber is thoroughly degassed. Thus nitrides of iron and aluminium have been formed by sputtering in a nitrogen atmosphere. Changes in the gas composition due to the presence in the chamber of vapours and gases liberated from internal surfaces may be prevented by either, or a combination of two ways :—

(i) exhausting the chamber with a vapour pump to a low pressure (about 1 micron Hg) to ensure thorough degassing prior to leaking-in the desired gas, or

(ii) exhausting the chamber with a pump possessing a sufficiently high speed at the sputtering pressure to permit a large leakage rate of the desired gas into the coating chamber, thereby ensuring that the pressure of the contaminant gases is a small fraction of that of the desired gas.

In a later section it is shown that both methods can be used if the plant is exhausted by a *booster vapour pump*, which has a high pumping speed at low and intermediate pressures.

REACTIVE SPUTTERING PLANT DESIGN

Successful reactive sputtering depends upon the maintenance of a stable glow discharge with the introduction of the required gas mixture into the work chamber at a constant rate under constant pumping conditions.

To maintain a glow discharge in a steady state, particularly when high current densities are used and an arc discharge may more easily develop, requires special h.t. power pack design if wasteful current-limiting resistances are to be avoided in the output circuit. Both metal oxides and metallic films may be sputtered rapidly, if high applied voltages and current densities are used, but because most of the power dissipated in a discharge is converted into heat energy it is sometimes necessary to water-cool the chamber and electrodes to prevent overheating of the vacuum seals and film support. Such water-cooling may influence the degree of oxidation and crystalline structure of the sputtered film, if these depend on the temperature reached by the film during deposition. With some metals the cathode may need to be cooled if only to prevent it from melting, *e.g.*, indium melts at 155°C and coatings of this metal on the surface of thick copper discs some 45 cm. in diameter have been easily melted at a power input of 3 kV and 0.3 mA/cm^2. The provision of a water-cooled cathode presents both vacuum sealing and insulation problems.

The principal problems associated with equipment design and control of reactive sputtering are discussed in detail below.

The Pumping System

Control of the Gas Pressure: The gas pressure, at which effective glow discharge conditions are obtained, is in the region 10–100 micron Hg (for

Vol
3
195

applied voltages between 1 kV and 10 kV). At these gas pressures a rotary mechanical pump has a very low pumping speed, and precise control of the power input to the glow discharge can be rendered difficult because the gas pressure rises uncontrollably due to outgassing. This difficulty can be overcome by exhausting the chamber with a vapour pump during sputtering, because at the operating pressure, the speed of vapour pumps is many times greater than that of single or two-stage rotary pumps of comparable cost and dimensions. The glow discharge pressure can then be controlled with an adequate supply of gas flowing into the chamber *via* a needle valve. A difficulty in operating such a system is that at the operating pressure, the *mass throughput* (generally measured in lμ/sec.) of a vapour pump remains fairly constant (see Fig. 2b) and the discharge gas pressure may vary within wide limits for very small changes in the rate at which gas is admitted to the system. If the vapour pump is fitted with a baffle-type high vacuum valve then it is possible to obtain a fine adjustment of the gas pressure by raising or lowering the valve plate thereby increasing or decreasing the valve aperture to control the pumping speed after a coarse pressure adjustment has been made with the needle valve.

Choice of Pump: As stated earlier it is necessary when preparing reactive sputtered films to a specified composition to eliminate the effects of residual gases either by evacuating the work chamber to an ultimate pressure lower than 0.1 micron Hg, or by having a very high pumping speed at the working pressure. A conventional vapour pump can be used for exhausting the sputtering chamber to low pressures (about 0.1 μ Hg) but it is not primarily designed to give high pumping speeds at the intermediate pressures used for sputtering, *i.e.* 10–100 micron Hg, nevertheless it can be used satisfactorily in small equipment. We have found in our laboratory that a 4-inch diameter vapour pump gives a good performance on a plant fitted with an 18 inch. diameter cathode.

In order to provide an adequate pumping speed at discharge pressures in large-capacity chambers it is desirable to use a booster vapour pump having a high-speed ejector stage with a critical backing pressure of about 3 mm. Hg. Such a pump should be fitted with a water-cooled high-vacuum baffle valve by means of which the pumping speed may be controlled in the manner described in the previous section. Continuous control of the pressure may be obtained by using a screw mechanism to raise the baffle plate.

A comparison of the characteristics of the pumps which have been mentioned will help to clarify the advantages of the booster type, and in Fig. 2 the *throughput* in lμ/sec. has been plotted against pressure for (a) a typical 9-inch. diameter booster pump (b) for a conventional 9-inch. diameter diffusion pump and (c) for a single-stage backing pump with a free air displacement of 450 l./sec. Both scales are logarithmic, and it can be seen that the booster pump has an effective pumping speed which is some five to ten times greater than the standard vapour pump in the pressure range 10–1000 micron Hg. Such a pump would provide satisfactory sputtering conditions in a plant fitted with a cathode 3 feet or more in diameter.

Gas Mixing Apparatus

For the deposition of metal oxides to a specified composition it is necessary to sputter in a gas atmosphere prepared by mixing oxygen with an inert gas in a fixed proportion. Very otten, the percentage of active gas required in the mixture is as low as 5%, and under these conditions a useful method of providing a given gas mixture is to have a reservoir in which the gases can be mixed before admission to the chamber, as shown in Fig. 1. The sputtering pressure is usually of the order of 10 micron Hg, and at an average pumping speed of 100 l./sec. the gas in a reservoir of a capacity of 20 litres at a pressure of one atmosphere will last for about four hours sputtering time. Over short sputtering periods, no adjustment of the needle valve is required, but as the pressure in the reservoir decreases, the needle valve must be progressively opened. A diagram of the pumping system and reservoir is shown in Fig. 1. The reservoir contains phosphorous-pentoxide trays which effectively remove any water vapour from the incoming gases. This is essential since water vapour is dissociated in the discharge and active oxygen and hydrogen are liberated, which change the composition of the carefully

prepared gas mixture.

At the beginning of a cycle, all the valves to the gas reservoir are closed, except for the diaphragm valve A which is opened to the chamber in order to exhaust the reservoir. The chamber and reservoir are pumped on the diffusion pump for at least ten minutes before the valve A is closed, so that all residual gases are removed. Less time is required using a booster vapour pump on account of its much higher pumping speed. Gases are then admitted from high pressure cylinders to the reservoir in proportion by pressure. Normally the gases are admitted until atmospheric pressure is reached, a relief valve being fitted to prevent excessive pressure rise. The needle valves C and D are used to control the gas input from the high-pressure cylinders to the reservoir, and it is important to ensure that there is no air in the lines between the reducing valves on the high pressure cylinders and the needle valves.

Cathode Cooling

Most of the electrical power supplied to the glow discharge is liberated as heat in the cathode potential fall and at least 50% of this is dissipated at the cathode. For rapid deposition of metal and oxide films very high power inputs are required and an input of 10–50 watt/sq. cm. of cathode surface is typical. When a low melting point metal is sputtered it is necessary to water-cool the cathode as shown in Fig. 1 to prevent melting of the cathode surface.

In one design of a water-cooled cathode used in our laboratory the h.t. connection to the cathode was made *via* the cooling water pipes which were brazed into a copper bush. This bush was sealed to a 'Perspex' insulating plug which was in turn sealed to the chamber by a rubber gasket. Six-foot lengths of 7 mm. internal diameter rubber hose were used to insulate the cathode from the inlet and outlet water mains, and the leakage current to earth was only 14 mA at 6 kV.

Controlled cooling of the receiving surface would be an advantage, since the surface temperature of the substrate controls the crystal formation. Also, it is known that the state of oxidation of a deposited film may be influenced by the substrate temperature. Thus cadmium oxide films tend to decompose as their temperature rises.

High Tension Power Supply

The d.c. supply for the glow discharge may be obtained from a single-phase bridge rectifier circuit where power outputs up to about 3 kW are required but, where larger power outputs must be used, a three-phase rectifier system is more practicable.

When high current densities are used, the transition from a glow to an arc discharge can more easily occur. Should the glow discharge temporarily pass from a limited area of the cathode, due to surface contaminants reducing the work function of a particular region, then appreciable local heating of the cathode may take place. This can give rise to the thermal emission of electrons which permits the transition from a glow to a high current arc discharge.

Thus it is necessary to limit the maximum output current to protect the rectifiers and transformers from overload and to prevent pitting of the cathode surface. The use of overcurrent relays in the h.t. power supply is not a complete solution to the problem because during a period of sparking from a contaminated cathode, the relays must be continuously reset. When voltage regulating auto-transformers are fitted in the primary circuit of the h.t. transformer to give stepless control of the output voltage, then overload protection may be required to protect the auto-transformer. A suitable overcurrent relay for this purpose is a motor starter fitted with oil dashpots so that only sustained sparking will throw out the contactor.

A current limiting resistor in the output circuit reduces the effects of arcing but to be effective the resistor must absorb considerable electrical power. Reactance limitation of the output current is the most efficient method of control. This may be provided by fitting chokes in series with the output load, or preferably by using a specially designed transformer with a secondary winding whose reactance rises as the load impedance falls, *i.e.* a transformer with exceptionally poor regulation. Such transformers have been used for many years for operating neon lighting.

The fitting of capacitors for smoothing purposes in

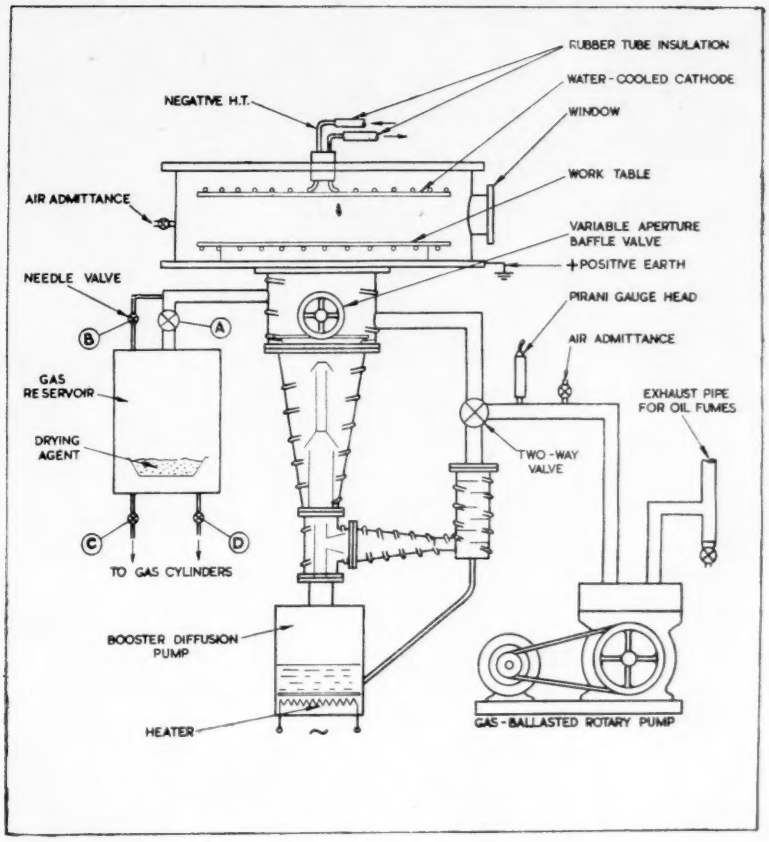


Fig. 1. A pumping system for reactive sputtering.

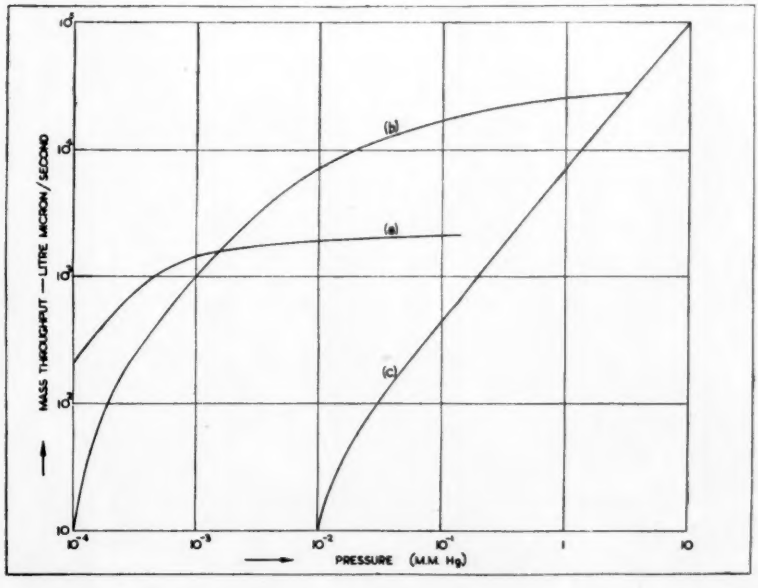


Fig. 2. Pumping characteristics of (a) a typical 9 inch. diffusion pump (Edwards 903B), (b) a 9 inch. booster pump (Edwards 9B3) and (c) a suitable backing pump with free air displacement of 450 l./min.

the output circuit of an h.t. power supply will also assist the formation of an arc discharge, because the capacitors will discharge considerable energy immediately there is any tendency towards the short circuit conditions of the arc discharge. Some metals, such as aluminium, form non-conducting oxide films on their surfaces and are prone to sparking when used as cathodes due to periodic electrical breakdown of the oxide barrier. The effects of smoothing capacitors and of insulating oxide films in promoting the formation of arc discharges has been described by Holland and Sutherland[19].

Acknowledgements are made to Mr. A. S. D. Barrett, Technical Director, Edwards High Vacuum Ltd., for his kind permission to publish this account.

References

1 OVERBECK, C. J., *J. Opt. Soc. Amer.*, **23,** (1933), 109. 'Colour in Films of Sputtered Tin'.

2 VESZI, G. A., *J. Brit. I.R.E.*, **13,** (1953), 183. 'The Modern Single Layer Selenium Photo-Electric Cell'.

3 GLOCKLER, G. and LIND, S. C., *The Electrochemistry of Gases and Other Dielectrics*, (Chapman & Hall Ltd., London, 1939).

4 PERROT, M., *Rev. Opt.*, **26,** (1947), 202-204. 'Augmentation des Facteurs de Réflection par une Couche Protectrice'.

5 KOENIG, H. and HEISINGER, L., *Festschrift Hereaus Platinschmelze*, (Hanau, 1947), 202. 'Sputtered Layers'.

6 BLEWETT, J. P. and JONES, E. J., *Phys. Rev.*, **50,** (1936), 464-468. 'Filament Sources of Positive Ions'.

7 PELCOWITCH, I., *Philips Res. Rep.* R.235, **9,** (1954), 42-79. 'A Study of the Evaporation Products of Alkaline Earth Oxides'.

8 HASS, G., *Vacuum*, **2,** (1952), 331-345. 'Preparation, Properties and Optical Applications of Thin Films of Titanium Dioxide'.

9 HOLLAND, L., HACKING, K. and PUTNER, T., *Vacuum*, **3,** (1953), 159-161. 'The Preparation of Titanium Dioxide Beam-Splitters of Large Surface Area'.

10 HAMMER, K., *Optik*, **3,** (1948), 495-498. 'Steigerung der Glas Reflection durch ein Metal Oxyd System'.

11 HOLLAND, L., HACKING, K. and MOUTOU, C., *Rev. Opt.*, **84,** (1952), 209-217. 'Optical Characteristics of Ion Oxide Films Deposited by the Evaporation Method'.

12 MCMASTERS, H. A., *Brit. Pat.* 632,256. 'Electrically Conducting Films and Method of Application'.

13 CUSACK, N. and BOWNESS, C., *J. Sci. Instrum.*, **31,** (1954), 345. 'The Use of Electrically Conducting Glass in Cloud Chamber Construction'.

14 AITCHISON, R. E., *Austral. J. Appl. Sci.*, **5,** (1954), 10-17. 'Transparent Semiconducting Oxide Films'.

15 PRESTON, J. S., *Proc. Roy. Soc.*, **202A,** (1950), 449-466. 'Constitution and Mechanism of the Selenium Rectifier Photocell'.

16 TAKEYA, K., SHIRAKAWA, J. and TAKAHASHI, F., *Le Vide*, **9,** (1954), 116-124. 'The Effect of Positive Ion Bombardment on the Oxide Cathode'.

17 HELWIG, G., *Z. Phys.*, **132,** (1952), 621-642. 'Electrical Conductivity and Structure of Sputtered Cadmium Oxide Layers'.

18 GOMER, R., *Rev. Sci. Instrum.*, **24,** (1953), 993. 'Preparation and Some Properties of Conducting Transparent Glass'.

19 HOLLAND, L. and SUTHERLAND, N., Edwards High Vacuum Ltd., *Information Sheet* No. E 161/1 (1955). 'Etching of Metal Specimens by Cathodic Sputtering'.

methods and techniques for

THE DETERMINATION OF SPECIFIC SURFACE BY GAS ADSORPTION

A. S. Joy, B.Sc., A.R.I.C.

Fuel Research Station,
Dept. of Scientific and Industrial Research,
*Greenwich, London, S.E.10**

Vol. 3 1953

Summary

A CRITICAL REVIEW is made of experimental procedures for the determination of specific surfaces and relative surface areas by the method of measuring the volume of gas adsorbed on the surface of the solid. Volumetric and gravimetric techniques are described and compared, and their suitability for routine and research purposes is indicated. The difficulties usually associated with the determination of gas adsorption isotherms are discussed, and a brief description is given of the methods used for calculating surface areas from the adsorption data.

Sommaire

REVUE CRITIQUE des procédés expérimentaux pour la détermination de surfaces spécifiques et de surfaces relatives par la méthode de mesure du volume de gaz adsorbé sur la surface du solide. L'on décrit des méthodes gravimétriques et volumétriques, elles sont comparées et leur utilité pour des buts de routine et de recherche est signalée. Les difficultées géneralement liées à la détermination des isothermes d'adsorption gazeuse sont discutées et il est donné une brève description des méthodes utilisées pour calculer les aires superficielles en partant des chiffres d'adsorption.

INTRODUCTION

SEVERAL METHODS are available for determining the size, shape, and distribution of size and shape of particulate solids. These data can be used to calculate a surface area which usually relates only to the ' smoothed ' external surface of the particle and gives little indication of the internal surface, particle, porosity and fine external structure. This apparent external surface area is sufficient for many purposes, such as the classification, and control during preparation of materials such as abrasives, cement, pulverised fuel, metal powders and some pigments, pharmaceuticals and fillers, but is of little value when internal surface and porosity are important in the use of the material. This is especially so for the very wide range of materials such as alumina-silica gel, kieselguhr, charcoals and carbon blacks, used as catalysts and adsorbents.

Furthermore, a knowledge of internal structure, external roughness, or porosity is frequently essential to the understanding of the fundamentals of surface phenomena. Gas adsorption methods, which depend on the measurement of the volume of gas adsorbed on the surface, are the most widely applicable means of obtaining such information, but some difficulty is usually experienced in choosing a suitable experimental technique and in interpreting the data. This is so mainly because papers dealing with techniques

* MS. received November, 1954.

are widely scattered throughout the literature, and because surface areas can be calculated by any one of the three following methods :—

(a) by applying the Brunauer, Emmett and Teller[1] equation
(b) by using the ' Point B ' method
(c) by measuring the volume adsorbed at some pre-determined pressure and applying an empirical conversion factor.

This review is mainly intended to cover the experimental techniques but a brief discussion of the above methods is given below for the sake of completeness :—

(a) The B.E.T. equation is

$$\frac{x}{V(1-x)} = \frac{1}{V_mC} + \frac{(C-1)x}{V_mC}$$

where x is the relative pressure, V is the volume of gas adsorbed, C is a constant for any given adsorbate/adsorbent pair, and V_m is the monolayer volume.

Several reviews of the uses and applicability of this and other adsorption isotherm equations have been published[1,2,3,4] and should be consulted for detailed information. Whenever applicable, the full equation, plotted from not less than three experimental points taken within the relative pressure range x=0.05 to 0.3, should be used when an accurate result is required, or when an entirely new type of material is being tested.

(b) If the first point of maximum curvature of a sigmoid isotherm is well-defined, then the ' Point B ' V_m value is very close to that obtained from the B.E.T. equation. The closeness of agreement is entirely a characteristic of the particular adsorbate/absorbent system, being dependent on the magnitude of the ' C ' constant,

GLOSSARY OF TERMS

Adsorbate. The gas being adsorbed.

Adsorbed Volume (V [ml.]). The volume of gas adsorbed at the pressure P.

Adsorbent. The solid on which the gas is being adsorbed.

B.E.T. Constant. The factor which relates the heat of adsorption to the heat of liquefaction of the adsorbate gas.

B.E.T. Plot. The straight line obtained by plotting $X/V(1-x)$ against x. The slope of this line is $(C-1)/V_mC$, and the intercept is $1/V_mC$.

Cross-Sectional Area (C.A.) The area occupied by one molecule of adsorbate on the surface of the adsorbent.

Dosing. Passing a known amount of gas from the gas burette into the sample bulb.

Equilibrium Adsorption Pressure (P [mm. Hg].) The vapour pressure of gas in equilibrium with the adsorbed liquid.

Hysteresis. Non-reversibility of the adsorption isotherm, *i.e.* the desorption isotherm follows a different path to the adsorption isotherm.

Knee. The first turning point of the isotherm.

Monolayer Capacity. The monolayer volume expressed either as the number of molecules or the weight of gas adsorbed.

Monolayer Volume (V_m [ml.]). The volume of gaseous adsorbate which forms a monomolecular layer over the surface of the adsorbent.

' n '. The number of adsorbed layers.

Point B. The first point of maximum curvature of a sigmoid isotherm.

Relative Humidity (R.H.). The customary manner of referring to the relative pressure when water is the adsorbate.

Relative Pressure ($x=P/P_0$). The ratio of the equilibrium adsorption pressure P, and the saturation vapour pressure P_0.

Saturation Vapour Pressure (P_0 [mm. Hg]). The vapour pressure of the bulk liquified adsorbate at the temperature of adsorption.

Sigmoid (or Type 2) Isotherm. The most commonly occurring S-shaped isotherm obtained by plotting V against P.

Specific Surface (A). The surface area per unit weight of adsorbent, usually expressed in sq. metres per gramme, but sometimes in acres per lb. (1 sq. m./g.=0.112 acres/lb.).

Surface Area (S). The total surface area of the adsorbent, obtained by converting V_m to the corresponding number of molecules and multiplying by the cross-sectional area.

Surface Packing Factor. The ratio of the number of adsorbed molecules actually required to cover unit surface area, to the number estimated from the known cross-sectional area of the adsorbate molecule.

and ' Point B ' should never be used unless its correctness for the system under consideration has been experimentally confirmed.

(c) Similar arguments apply to the methods of the third type, *i.e.* : the use of various semi-empirical and ' single isotherm-point ' methods, even when these are used only for batch or sample comparison and not for the determination of an absolute surface area value. Single or two-point methods should be checked, at least once, by comparison with the B.E.T. method. Even if the results agree the following observations should be noted :—

(1) If the ' C ' value of any two samples is the same, then the ratio of the volumes adsorbed at any pressure is the same as the ratio of the surface areas.

(2) If the ' C ' values, also, are different, then the volume and area ratios are no longer the same, although the two isotherms may cross each other once or even twice.

(3) The slope past the ' knee ' of a normal sigmoid isotherm is usually small, so that V is insensitive to small changes in P or x and depends mainly on V_m.

According to (1) and (3) the comparison is best made by noting the volumes adsorbed at the same predetermined pressure. On the other hand (2) implies that comparisons should be made at low pressures where the slope is large and mainly dependent on ' C '. In view of the consequent uncertainty in the interpretation of a single measurement it should only be used when the speed of measurement is more important than the accuracy of the result. The uncertainty is considerably reduced if experiments show that the heat of adsorption is sensibly constant from sample to sample and batch to batch of the same material.

GENERAL OBSERVATIONS ON EXPERIMENTAL TECHNIQUES

Degassing

It is clear that any adventitious gas already adsorbed on the surface has to be removed before quantitative measurements can be made of the total amount adsorbed. What is not so obvious is the extent to which this cleaning or degassing process need be carried out. For more accurate work it is necessary to use the most drastic means permissible without causing decomposition, sintering or transformation of the material; the operating conditions should be, as far as possible, identical in each series of experiments.

The latter stipulation should be even more rigorously adhered to in test and control work where the degassing conditions have been chosen empirically on the grounds of expediency. Some guidance in the choice of conditions is given by the following observations largely based on the writer's own experience :—

Adsorbed Impurities. Strongly adsorbed impurities do not usually affect the measurements but all or any impurities present in sufficient amounts to fill or block pores or capillaries will seriously reduce the measured surface area. If the ratio T_b/T_a* is greater than 3 then the presence of the impurity does not affect the B.E.T. surface area, but may alter the heat of adsorption. If the ratio is less than 3, the adsorbed impurity will probably affect both, V_m and ' C '.

Pressure. With the pump running, the pressure in the adsorbent bulb during degassing should be less than 10^{-4} mm. Hg. Under this condition the rate of degassing is largely controlled by diffusion from the interior of the particle.

Temperature. An example of the interrelation of temperature and time to achieve the same state of degassing is shown in Table 1 below :—

Table I

The Effect of Temperature on the Degassing of Coke

Temperature °C	*Time hr.*	*Temperature* °C	*Time hr.*
100	36	300	6
150	26	350	4
200	18	400	3
250	12	450	3

The form of the time/temperature relationship of Table 1 is general but the magnitudes are not. At any

* Where T_b is the boiling point of an adsorbed gaseous impurity and T_a is the temperature of adsorption measurement in °K.

given temperature the degassing time decreases with decreasing capillary length and increasing pore radius, and ultimately a non-porous surface is readily degassed by heating at 350°C for 15 minutes.

Final State. An acceptable ' standard degassed state ' of the adsorbent is reached when the sample is evolving 2×10^{-10} ml. of gas per minute per square cm. of surface. This rate of vapour evolution in a normal system shut off from the pumps and cold trap gives a pressure of 5×10^{-4} mm. Hg after 15 minutes. The rate may be increased tenfold if the required accuracy is low, and may also be allowed to vary within fivefold limits during routine batch or sample comparisons. The pressure with pumps running and cold-trap in position should on no account be quoted as a criterion, although it may be used for routine work on the same material if experiment has shown that it can be related to the standard state of degassing.

Quick Method. The time taken to degas a sample can often be very considerably reduced by ' washing ' the sample with the adsorbate vapour. This effect was observed by the writer when carrying out a series of replicate experiments on the same sample and it was confirmed that there was no change in the measured surface area. It has since been noted that McBain[58] used this procedure and strongly recommended other workers to adopt its use. The sample bulb is evacuated for a few minutes at the degassing temperature, and subsequently shut off from the pumps and cooled to the adsorption temperature. The adsorbate gas is then admitted to the bulb, until a pressure of a few cm. Hg is reached, no measurement being made of this quantity. The adsorbed gas is then pumped out, firstly at the adsorption temperature, then at room temperature, and finally at the degassing temperature. As a consequence of this cycle of operations, the time needed to obtain the standard degassed state should be reduced to about one-third of the ordinary value.

Sample Entrainment. If the sample is a very finely divided or light powder, it is very readily entrained in the gas stream during evacuation and degassing. This ' fluidisation ' phenomenon is most marked when the tap between the sample bulb and the vacuum manifold is opened at the start of the evacuation. The operation should therefore be performed slowly and with great care. Several attempts have been made to minimise this effect, the most satisfactory of which is the inclusion of a small plug of glass wool in the tube just above the sample. This prevents gross carry-over if the tap is opened too quickly, but the plug can become choked with powder and cause all gas transfer processes to be extremely slow. In such a case it is quicker and safer to restart the experiment.

Some sample-bulbs are designed with top and bottom exit tubes, and this certainly reduces the risk of initial fluidisation but is no safeguard against the sudden evolution of desorbed gases which may take place during hot degassing. Silica gels and adsorbent charcoals are particularly prone to exhibit the latter effect and in a difficult case the only real cure is to be extremely careful when evacuating, heating and degassing the sample. Nevertheless, some (often almost invisible) quantity of powder is swept out of the bulb and settles in the connecting tubing. The accumulation of material after several experiments will gradually foul the apparatus, so that it is not advisable to degas through the burette and manometer system but to provide an alternative path straight to the vacuum manifold. This will also reduce the length of capillary tubing through which the sample has to be evacuated, and thereby ensure that a pumping speed faster than the rate of surface desorption can be maintained in the bulb.

Most of the above remarks apply only to volumetric apparatus operating at pressures between 10 and 600 mm. Hg, in which small bore tubing is used to reduce the volume of the burette manifold and connecting lines. Low pressure and gravimetric systems have wide bore connecting tubing in which fluidisation and pumping difficulties are not usually encountered because the sample can be spread loosely in a wide tube or bucket. Furthermore, the large volume of these systems means that it is comparatively easy to reduce the pressure slowly.

The Magnitude of the Surface Area

The capabilities of any particular gas adsorption apparatus should not be assessed in terms of the smallest and largest specific surfaces which it can

Vol
3
195

measure, but rather in terms of the total surface area expressed as the product of the specific surface and the weight of the sample which can be accommodated in the sample-bulb. In other words, the limits are dependent as much on the density of the material and the volume of the sample-bulb as on the specific surface of the adsorbent. However, the volume of the sample-bulb and of the tubing connecting it to the burette manifold should be kept as small as possible, and it is recommended that this (dead space) volume should not be larger than 40% of the expected monolayer volume.

These comments refer mainly to the volumetric technique, but similar remarks apply to the gravimetric method in which the sensitivity and loading limits control the range of the apparatus. Once a required range of surface area measurements has been decided upon, very little difficulty should be experienced in choosing an adsorption apparatus to cover this range. In volumetric research work, a multi-bulb two-manometer apparatus can cover a range of from 2 to 800 m^2, but smaller areas and routine operation demand specially designed equipment. A standard gravimetric apparatus, working on the spring balance principle, can be made to cover the entire range from 0.1—1000 m^2, in three steps of about 0.1—10, 10—400 and 400—1000 m^2 simply by changing the spring.

The Adsorbate Gas

The two main requirements of the adsorbate gas in physical adsorption are: (a) It should not react with the adsorbent or any adsorbent constituent (b) it should be readily obtainable in a high degree of purity.

In principle, any gas which fulfils these conditions may be used; however, the gas may be unsatisfactory for other reasons, *i.e.* too low or too high a vapour pressure, low heat of adsorption or variable surface packing factor. The best adsorbates are those given below :—

— nitrogen,
— the rare gases,
— halogenated methanes,
— methyl and ethyl alcohols,
— water.

Water is sometimes anomalous in its behaviour, *e.g.* on graphite, but it is often very satisfactory especially if used in a gravimetric apparatus.

The Temperature of Adsorption

The choice of temperature is controlled by three considerations :—

— the nature of the adsorbate gas,
— the lowest temperature to which the adsorbent can be taken without undergoing some physical change and
— the ease of preparation, constancy, etc. of the refrigerant bath.

The first refers to the vapour pressure of the adsorbate at the temperature of adsorption. Its value should be such that a simple method can be used for the pressure observations. There is basically no objection to using a gas at temperatures far below its normal boiling point and reading with a McLeod gauge, or at temperatures far above its boiling point and reading with a dead-weight gauge. These methods should however only be considered in special circumstances. The pressures usually encountered are between 5 and 1,000 mm. Hg and are read with fluid-filled manometers.

The second applies mainly to organic adsorbents containing entrapped fluids which may freeze and disrupt the structure, but some inorganic substances may undergo phase transformation or rearrangement phenomena. The effect of ordinary thermal contraction can be ignored, but the anomalous contraction of plastic and resinous materials can cause considerable deformation of shape and structure.

Regarding the third, satisfactory baths can be made from Dewar flasks containing melting solids, eutectic mixtures and boiling liquids, the bath temperature increasing roughly in that order. The most common gases and refrigerant-baths in the order of preference are :—

(1) Nitrogen gas, and liquid nitrogen or liquid oxygen at −195 or −183°C.
(2) Dichlorodifluoromethane (CF_2Cl_2), known as Freon, and magnesium chloride ice eutectic (equal weights of $MgCl_2.6H_2O$ and water

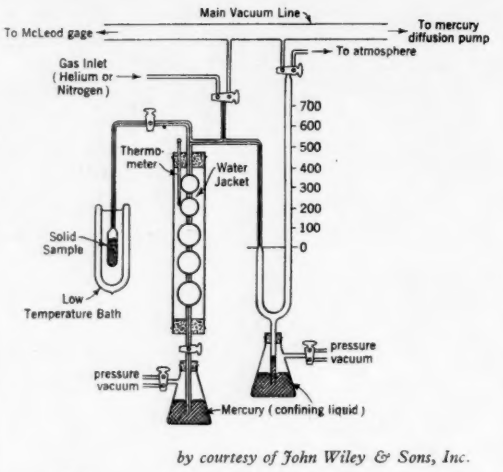


Fig. 1. The prototype of most modern designs. The essential components are a multi-bulb burette, a manometer and a sample bulb.

cooled with powdered solid CO_2) at –33.1°C.

(3) Water vapour in equilibrium with solutions of known vapour pressure, and a water- or air-bath at 25°C.

(4) Ethyl chloride, and ice at 0°C.

(5) Carbon tetrachloride, and a water- or air-bath at 25°C.

The apparent cross-sectional area of an adsorbed molecule varies more rapidly with temperature than predicted by the change in liquid density. This appears to be due to an alteration in the packing factor of the molecules on the surface, probably caused by the resemblance of the adsorbed layer to a two-dimensional anisotropic gas. Surface areas calculated from isotherms obtained at appreciably different temperatures, even for the same adsorbate, should therefore be compared with caution.

The Choice of Technique and Apparatus

The fact that many workers prefer, or find it necessary, to design their own apparatus is sufficient proof that it is impossible to give advice which would satisfactorily cover all needs and circumstances. This is mainly due to the diversity of reasons for constructing an apparatus and to the different conditions under which it is to be used. However, all but a few of the methods employed can be assigned to one or the other of two main classes, *i.e.* volumetric or gravimetric methods depending on whether the volume or the weight of gas adsorbed is observed in the experiment. Each of these classes may be further, but not so accurately, sub-divided into basic types of apparatus and their modifications for special purposes.

This general classification has been adhered to for the purpose of the present review. A more detailed analysis is given in the introductory notes to the appended bibliography for the guidance of readers who wish to study the subject or some particular aspect of it more closely.

EXPERIMENTAL TECHNIQUES—VOLUMETRIC

Conventional Apparatus

The so-called conventional adsorption apparatus invariably follows the standard assembly originally described by Emmett,[11] shown in Fig. 1 upon which a large number of workers have based their own apparatus. Adsorbate gas is taken into the burette and its pressure read on the manometer. The stopcock between the sample and burette is then opened, and the new pressure, after allowing time for equilibrium to be established, is read on the manometer. The volume of gas admitted to the sample bulb is proportional to the difference in the pressures before and after opening the stopcock; this latter pressure is also the equilibrium adsorption pressure. The volume adsorbed is equal to the volume admitted less the volume of gas required to fill the free volume (dead space) in the sample bulb and burette connections, and in conjunction with the equilibrium pressure value gives a single adsorption point. Further points can be obtained by closing the sample stopcock, admitting more gas to the burette, noting the pressure on the manometer, and then re-opening the sample stopcock. The apparent dead space at

each pressure is readily obtained by repeating the experiment with a relatively non-adsorbable gas, such as helium.

A disadvantage of this adsorption procedure is that the total volume admitted is found by summing the separate doses, so that errors are cumulative.

Emmett and Brunauer[8] describe an apparatus (see Fig. 2.) for the preparation and investigation of catalysts which by simple modification[9] reduces to the basic apparatus of Fig. 1, the application of which has been explained and discussed by Emmett in a number of publications[10,11,12,13].

The paper presented to the A.S.T.M. Symposium[13] is the source of the often quoted statement that Emmett claims an accuracy of only about 20% for the method. Emmett's comment actually concerned the uncertainty in the choice of molecular cross-sectional area for nitrogen, which gives about a 20% difference depending on whether 13.8 or 16.2 Å² is used. The modern figure for adsorption is 15.8 Å² with an uncertainty of only ±3%.

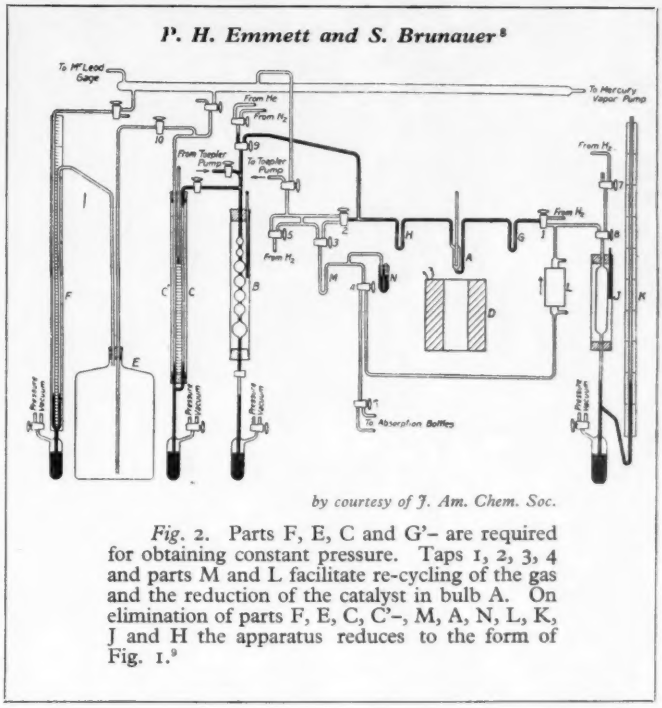


Fig. 2. Parts F, E, C and G'– are required for obtaining constant pressure. Taps 1, 2, 3, 4 and parts M and L facilitate re-cycling of the gas and the reduction of the catalyst in bulb A. On elimination of parts F, E, C, C'–, M, A, N, L, K, J and H the apparatus reduces to the form of Fig. 1.[9]

Joyner[14] gives a full account of the basic principles, and a step-by-step description of the construction and assembly of a complete apparatus. The fundamental system is conventional (Fig. 3) and the final assembly shown in Fig. 4 represents a typical all-glass modern apparatus. A summarised account of this design has also been given by the same author[15].

A similar apparatus[16] designed for greater accuracy is shown in Fig. 5. It embodies a built-in vapour pressure thermometer and a pair of differential manometers. Capillary sticking-friction effects are often observed in manometer A but these can be minimised by always reading at the null point.

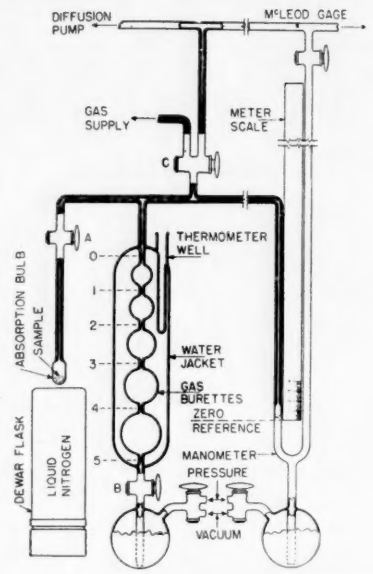
Ries, van Nordstrand and Teter[17] use the conventional system for the adsorption of nitrogen on materials with specific surfaces of between 10 and 200 m². The apparatus is designed for routine operation, and informative discussions of equilibrium time, dead space calculations, etc. are given in their paper. This type of semi-routine conventional apparatus has been used for correlating the surface areas of carbon blacks with other properties such as nigrometer values[18], and for testing metal powders[19]. The latter papers have been quoted because they give an excellent account of the difficulties likely to be experienced, and at the same time survey the means of avoiding or minimising these troubles.

Apparatus for Measuring Small Adsorptions

Very low pressure adsorption is in many respects simpler to measure than adsorption at higher pressures. All tubing can be large-bore, since the S.T.P. volume of gas contained in it at a pressure of a few hundredths of a mm. Hg is negligible.[25] This means that the dead space volume can safely be neglected provided the total adsorption is greater than 1 or 2 ml.[26,30,31].

If the surface available is very small, and the

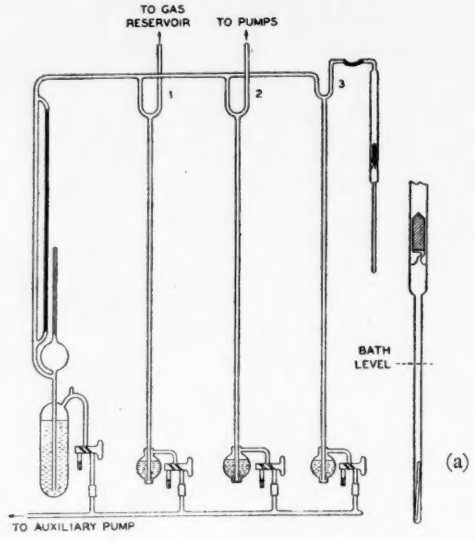
W. E. Barr and V. J. Anhorn [15]



by courtesy of Instrument Publishing Inc.

Fig. 3. This is a typical modern form of the construction shown in Fig. 1.

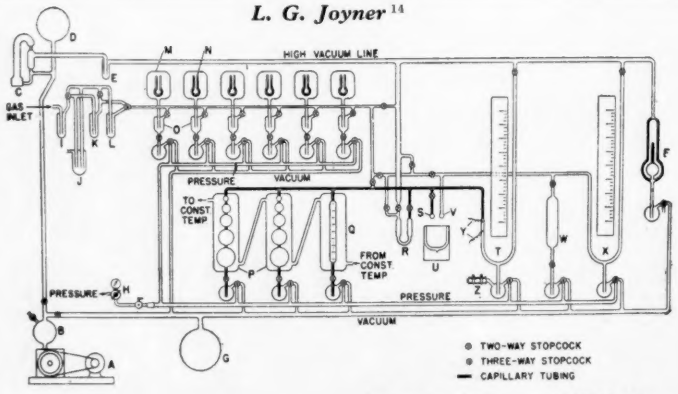
L. A. Wooten and C. Brown [26]



by courtesy of J. Am. Chem. Soc.

Fig. 6. Low pressure adsorption apparatus. (6a) Adsorption chamber containing cathode demounted from experimental vacuum tube.

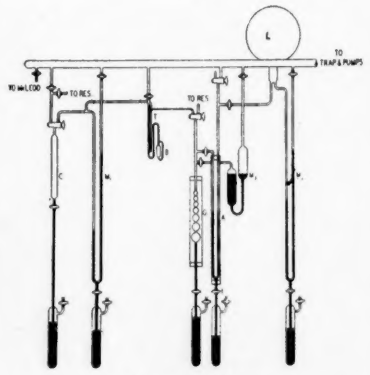
L. G. Joyner [14]



by courtesy of Instrument Publishing Inc.

Fig. 4. This apparatus incorporates the assembly of Fig. 3. P and O are multiple burette systems, B and W are built-in vapour pressure thermometers, X and R are differential manometers and Z and Y are the fine control and electrical zero devices. A–F and I–O are the pumping and gas storage systems respectively.

W. D. Harkins and G. Jura [16]



by courtesy of J. Am. Chem. Soc.

Fig. 5. This is the same basic skeleton as Fig. 4, but includes two extra differential thermometers. Manometer M3 is a low pressure manometer of wide bore 25 mm. tubing, to reduce errors in pressure readings due to surface tension. Capillary manometer A is to reduce the errors in volume readings due to levelling at high pressures. It is levelled by adjusting the pressures in the 5 l. bulb L until there is zero differential and the pressure is then read on manometer M_2.

Vol. 3 1953

pressure higher than 10 mm. Hg, measurement of the gas dosed into the sample bulb and of the dead space becomes very important. The usual method of achieving this purpose is to use a ' displacement doser ' technique, in which a very small volume of gas is completely displaced by mercury from a calibrated tube into the sample bulb. Such an apparatus is commonly known as the ' Tompkins and Young apparatus ' which, as first described[20,21] consisted of a displacement doser of about 15 ml. capacity and a differential oil manometer, reading up to 15 mm. Hg pressure. The most important modification[22,23] has been the introduction of a graduated burette and sintered-disc doser system enabling more points to be taken with greater speed of operation. Apart from the complete displacement principle, it is essentially a ' micro-conventional ' apparatus, and cannot deal easily with adsorption at pressures above 20 mm. Hg, on surfaces smaller than $0.01 m^2$, for which purpose[20] the differential method of Orr[24] is still the most satisfactory.

In Orr's apparatus the dead space correction is eliminated by using two identical adsorption systems, the volumes of which are arranged to agree as closely as possible. Adsorbent is put in one of the sample bulbs and the other is left empty, so that, if equal volumes of gas are admitted to the two bulbs, the change in pressure, read on a differential manometer connected between the bulbs, is a measure of the volume adsorbed.

Adsorption at very low pressures on very small surface areas can be readily measured by means of standard high vacuum measuring techniques[25,26] using wide manifolds, mercury cut-offs instead of taps and McLeod gauges instead of manometers.

The simplicity of the technique led Wooten and Brown[26] to choose it for measuring the specific surface of cathodes covered with spongy oxide layers. Their apparatus shown in Fig. 6 is typical of its kind, and the authors were able to measure the adsorption of 10 ml. (S.T.P.) of ethylene at a pressure of about 5 micron Hg corresponding to a relative pressure of $x=0.5$. This apparatus was closely copied for the study of the corrosion of aluminium foil[27], the main difference being in the method of correcting for temperature-pressure gradients.

The plain wide-bore manifold and cut-off system was also used by Armbruster and Austin[28], with the later addition of a double-armed McLeod gauge, and an improvement in the ratio of the adsorption bulb volume to the total volume[29]. Gans, Brooks and Boyd[30] used a fixed single bulb doser, with a wide manifold and a dibutyl phthalate manometer and concluded that such a low (1–10 mm. Hg) pressure apparatus could be satisfactorily used with water, propyl alcohol and benzene at room temperature.

A semi-routine method[31] has been devised for the measurement of the small surface areas presented by drawn glass fibres. A standard apparatus was modified to permit nitrogen adsorption isotherm points to be obtained at three predetermined pressures. A straight burette of 50 ml. capacity subdivided in 0.1 ml. was used in conjunction with a U-tube manometer, and the gas pressure kept constant by continuously raising the mercury in the gas burette. The authors correctly point out that once a fibre type has been classified from a complete run, it is only necessary to determine the dead space and the volume adsorbed at one pressure, for further routine testing. It is claimed that these two measurements take less than one hour.

Harkins and Jura[32] employed a simple wide-bore manometer and burette assembly for measuring adsorptions at pressures below 100 mm. Hg.

A totally different approach was used by Bangham and Mosallam[33] for studying the adsorption of organic vapours on mica. Their problem was complicated by the need to maintain the entire adsorption apparatus at temperatures of between 20 and 50°C, whilst measuring the adsorption of only a few ml. of gas at pressures of about 15 or 20 cm. Hg. Their apparatus is shown in Fig. 7. It will be noted that it is completely immersed in the bath and operated entirely from above the bath by means of the taps K. This apparatus should be compared with that of Harris and Emmett[44] described below (see page 265) which is superior in some respects.

Modifications of the Conventional Technique

As before the apparatus described in this section

D. H. Bangham and S. Mosallam[33]

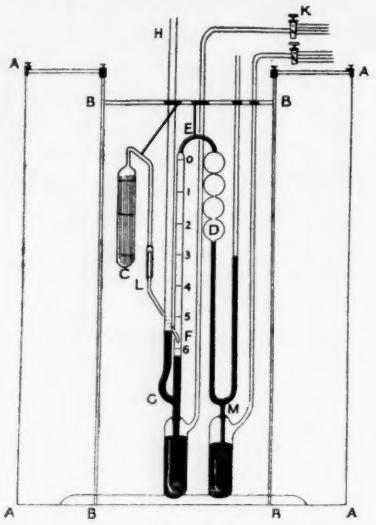
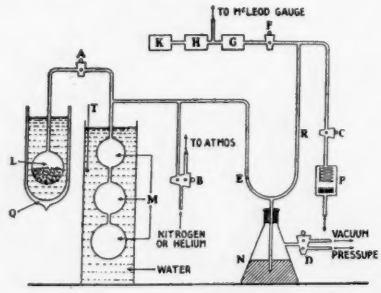


Fig. 7. The adsorption bulb is isolated from the manometer H and inverted U-burette FED, by raising the level of the mercury to above the insert tube at F until the float-valve L closes. Vapour is admitted to the burette through the cut-off M, levelled in DEF, its pressure measured on H, and finally dosed into the sample bulb by lowering the mercury level below F. The dead-space measured with helium in the usual manner is the volume of C up to the valve L.

P. E. Bugge and R. H. Kerlogue[35]



by courtesy of J. Soc. Chem. Ind.

Fig. 8. Apparatus for determining surface area by low temperature adsorption isotherms, A, B, C, D, F : Vacuum taps, greased with high quality vacuum grease. L : Sample bulb, volume usually about 5 c.c. Q : Dewar flask containing liquid nitrogen. M : Water-jacketed bulbs, total volume about 145 c.c. T : Thermometer. R : Manometer, length of R.H. limb, 60 cm., with mark E etched on L.H. limb. N : Manometer reservoir, 500 c.c. capacity, containing mercury. P : Drying train (phosphorus pentoxide and calcium chloride). G : Trap, immersed in dry ice and acetone. H : Diffusion pump. K : Rotary oil (backing) pump.

are all based on Emmett's design (Fig. 1) but differ from his in points of construction or mode of operation.

The usual variation dispenses with the multi-bulb burette and uses a single bulb of fixed volume as a reservoir or dosing chamber. This greatly simplifies the construction and operation of the apparatus, but considerably reduces the versatility of the method.

One of the earliest of these ' elementary ' pieces of equipment, intended primarily for single-point working, is that of Bugge and Kerlogue[34] (see Fig. 8). The diagram is self-explanatory, but it should be noted that the sample is degassed through the whole length of the adsorption manifold and manometer. This is often unsatisfactory for the reasons given earlier in this review (see page 257 : *Sample Entrainment*).

Fixed-volume bulbs confine the use of the apparatus to samples which do not vary much in total surface area, and the large burette volume used by the authors limits the useful range to between 100 and 300 m.^{2}. The same authors[35] give a simplified method of calculation which eliminates the dead-space determination, if two or more adsorption points are taken. It appears that this procedure is satisfactory only when the product V_mC is large enough to cause the B.E.T. plot to pass nearly through the origin.

A similar apparatus for routine testing of cracking-catalysts is described by Salvi and Fiumara[36] in a review (in Italian) covering theoretical principles, experimental techniques and hypotheses on the nature of adsorption phenomena. Their apparatus differs from that of Fig. 8, only in that it has ' magic-eye ' electrical zero indication, fine control squeeze tubes and a built-in vapour pressure thermometer similar to those described by Joyner[14].

The greatest simplification consistent with reasonable accuracy has been achieved by Smith[37] who uses

Vol. 3 1953

an ordinary, Fortin-type barometer tube to measure the pressure, a single 100 ml. bulb as a doser and gas reservoir, and a separate stopcock inserted between the sample and the vacuum manifold for degassing. Only single-point, or two-point determinations are made and the area values are read off directly from a chart prepared by calibrating the apparatus with samples of known specific surface.

Deitz and Gleysteen[38] designed an apparatus to reduce degassing times and speed-up experimental procedures. As can be seen from Fig. 9, the main apparatus was conventional, but four stopcock and taper joint units were fused to the adsorption manifold. All four sample bulbs were degassed at the same time in a large furnace at 400° C for 18 to 24 hours, and adsorption was carried out using each bulb in turn. The adsorbent-bulb was rather complex, with top and bottom exits, and a U-bend trap in the inlet tube.

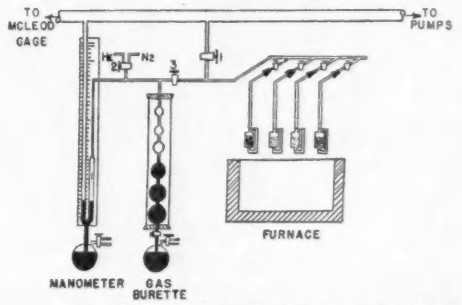
The apparatus in use at the Fuel Research Station for the investigation of the properties of coals, cokes and Fischer-Tropsch catalysts is essentially conventional but incorporates an extra manometer : M_2 (see Fig. 10). This manometer is used solely for reading the equilibrium pressure, and enables a large number of isotherm points to be obtained from a single filling of the burette, thereby avoiding cumulative errors. With minimum and maximum burette volumes of 10 and 150 ml. respectively, isotherms up to $x = 0.8$ can be obtained by a single filling for total surface areas of 2-200 m^2. Areas up to 1000 m^2. can be handled by two, or at the most three, burette fillings. The manometer tubes are backed by glass mirror scales, enabling the mercury levels to be read to ± 0.1 mm. without parallax, but pressures below 10 mm. Hg are read to ± 0.01 mm. with a cathetometer. For accurate work, a new sample bulb should be fused onto the adsorption manifold for each determination, but A7 taper joints can be employed for routine estimations, or when ' filler-plug ' and Anderson pattern[43] bulbs are used.

Fig. 11 is a photograph of this apparatus, taken before the electrical zero contacts were added.

Another method of refining the conventional apparatus is to use wide-bore manometer tubes, fitted with vernier sliding scales after the style of the ordinary standard barometer[40]. By this means the pressure can be easily and accurately read to 0.1 mm. Hg. If a micro-cathetometer is used it can be read to 0.01 mm. Hg. Incidentally, this paper[40] is well-known for the description of a method of applying the B.E.T. equation, including the parameter n, to all adsorption isotherms.

The apparatus of Krieger[41] (Fig. 12) is the simplest of the medium-accuracy fast-operating apparatus, and is highly recommended in its original (or in a modified form[42]). After degassing, the sequence of operation is to close Tap 1, fill the bulbs with gas and

V. R. Deitz and L. F. Gleysteen[38]



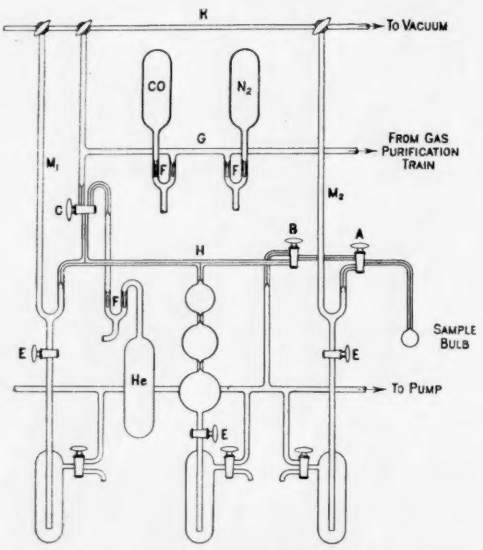
by courtesy of National Bureau of Standards

Fig. 9. This apparatus enables several sets of experiments to be prepared at the same time.

raise the mercury to the mark below B4, with E evacuated. Stopcock 1 is then opened, and the level re-adjusted to the original zero mark. Subsequent points on the isotherm are then obtained merely by raising the mercury level to the successive graduation marks between the bulbs, and noting the height of the mercury in the right-hand limb. The tubing between the bulbs is the same bore as in the other limb so that if the heights of the successive marks are known, there is no need to re-zero the manometer for any points after the first. This eliminates the usual trouble of admitting successive doses (and thereby avoids cumulative errors) and also effects a considerable saving of time whatever kind of multi-bulb

apparatus (for instance Figs. 1, 3, 9 and 10) is employed. Even, if the manometer should have to be re-levelled after each change of burette volume the total number of operations is reduced because the tap to the sample bulb is opened only once and the burette is filled with gas only once in order to obtain five or six points on the isotherm.

Fuel Research Station[39]



Crown Copyright Reserved

Fig. 10. Schematic representation. Taps A, B and C : Hollow key, 2-way vacuum stopcocks, 2 mm. bore capillary arms. Taps E : Hollow-key, straight-through, spring-loaded vacuum stopcocks, 3 mm. bore of key, 8 mm. bore side-arms. F : Mercury float-valves, operated manually by mercury reservoirs (not shown). M_1 and M_2 : 8 mm. bore manometer tubes, 100 cm. long. G : Gas reservoir manifold (4 mm. i.d.). K : High vacuum manifold (10 mm. i.d.).

The method has the disadvantage that the position of the isotherm points cannot always be regulated to fall conveniently within the B.E.T. range ($x = 0.05$ to 0.3). This can be partially offset by selecting the number of bulbs and their sizes to cover the expected range of adsorbed gas volume. In any case the versatility of a multi-bulb apparatus must always be greater than that of a single-bulb apparatus.

Gregg and Sing[42] modified Krieger's apparatus by using a continuously graduated burette fused to a plain tube of the same bore, instead of the bulbous-limbed U–tube of Krieger. The advantage of this modification is that a very large number of points can be taken between the upper and lower limits of the burette.

Original Designs and Special Methods

Harris and Emmett[44] have modified a conventional apparatus in order to investigate the adsorption of vapours above 75°C as shown in Fig. 13. The apparatus closely resembles that of Bangham and Mosallam (Fig. 7) but can deal with much larger gas volumes and pressures. Its main features are an ingenious cut-off between the sample bulb S and the burette, and the sealed-in adsorbate ampoule. All tubing external to the bath is wrapped with electrical heating tapes.

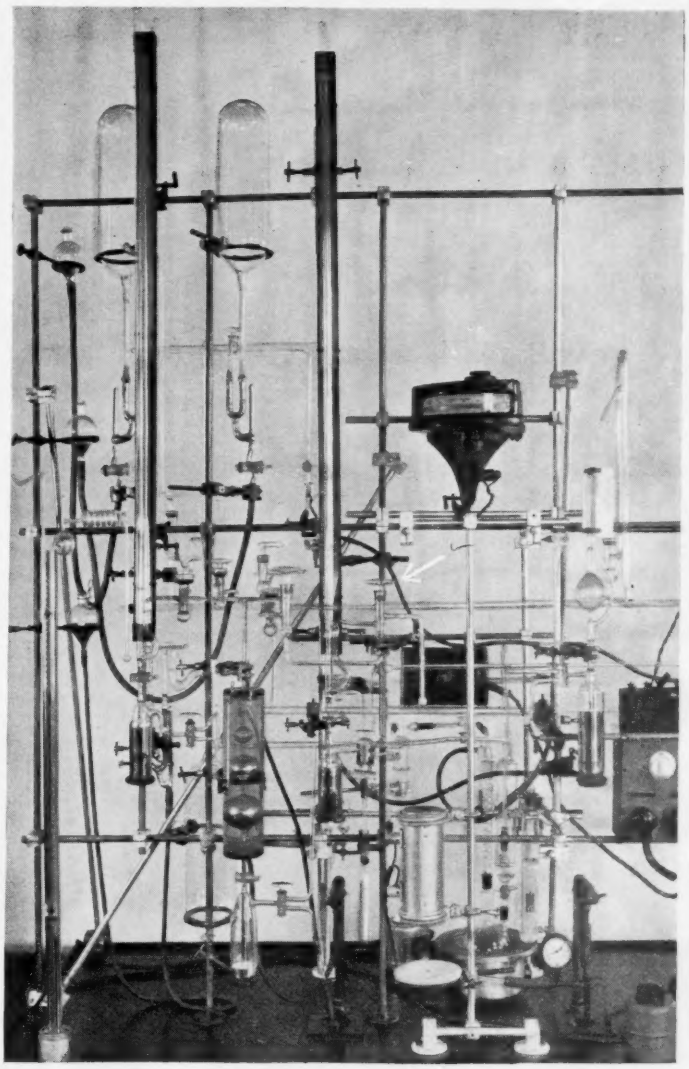
A fairly robust and relatively inexpensive apparatus with a number of unique features designed by Askey and Feachem[45] in 1938 is shown in Fig. 14. After slight modifications the burette and double manometer assembly would closely resemble that of Fig. 10. The adsorption bulb manometer is levelled against a Torricellian vacuum and the U–tube burette-manometer is levelled against atmospheric pressure. There is little objection to the former procedure, but the latter involves an extra correction to standard pressure for each reading. Unfortunately this cannot be remedied without losing the basic simplicity of the method. Furthermore, the manual lifts should be fitted with bubble-traps where the rubber tubing joins the manometers.

In industry rapid and simple methods are needed for the comparison of batch samples, and the Shell Group[46] have devised such a method, see Fig. 15. The specific surfaces are calculated by means of an empirical equation based on a calibration of the apparatus with samples of known surface area. The comments on page 256, paragraph (c), are especially applicable to this type of estimation.

To avoid the use of high vacuum techniques and to simplify the construction, Loebenstein and Deitz[47] adopted an entirely new approach, see Fig. 16. The conventional degassing process was avoided by heating the sample in a slow stream of pure helium, a procedure which must cast some doubt on the extent of degassing, although the authors' results appear to be satisfactory. In general the method is crude and the results are not very easily worked out, but it has the outstanding merit of being the only truly portable apparatus yet designed.

The arrangement[48] of Fig. 17 has the merit of extreme simplicity of design and operation. Anyone intending to carry out rough routine sample comparisons would be well advised to ascertain whether it is sufficiently accurate for his purpose. It can be used to compare two samples directly, or, after calibration, to obtain ' absolute ' values for the quantity of water adsorbed by the material at a known relative humidity. Smith and Green who devised the method attempt to calculate the specific surface by assuming that only a monolayer of water is adsorbed in their experimental procedure. With a flask volume of 1.2 l. and a manometer sensitivity of 0.00714 mm. Hg per mm. Hg they find that the specific surface is equal to 0.028 times the differential pressure reading. Calculation shows that the monolayer condition is nearly fulfilled for samples of total surface area of between 3 and 30 m.² and a relative humidity of 0.03 at 25°C. If reproducible results are to be obtained

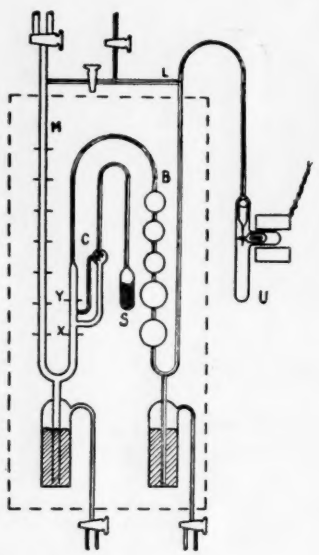
Fuel Research Station[39]



Crown Copyright Reserved

Fig. 11. Photograph of the apparatus of Fig. 10. The arrow indicates the 2-way dosing and manometer stopcock ' A ' of Fig. 10.

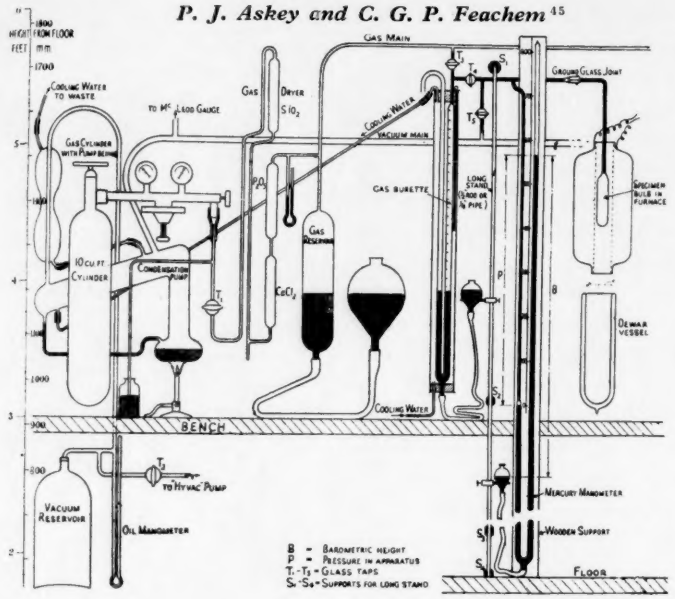
B. L. Harris and P. H. Emmett [44]



by courtesy of J. Phys. Chem.

Fig. 13

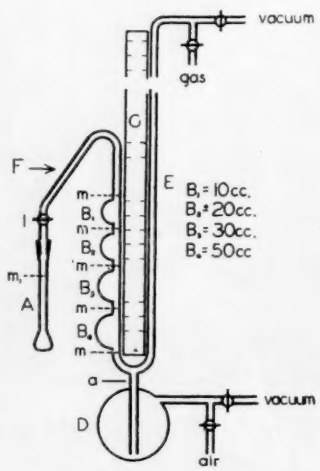
P. J. Askey and C. G. P. Feachem [45]



by courtesy of J. Soc. Chem. Ind.

Fig. 14

K. A. Krieger [41]



by courtesy of Industr. Engng. Chem.

Fig. 12

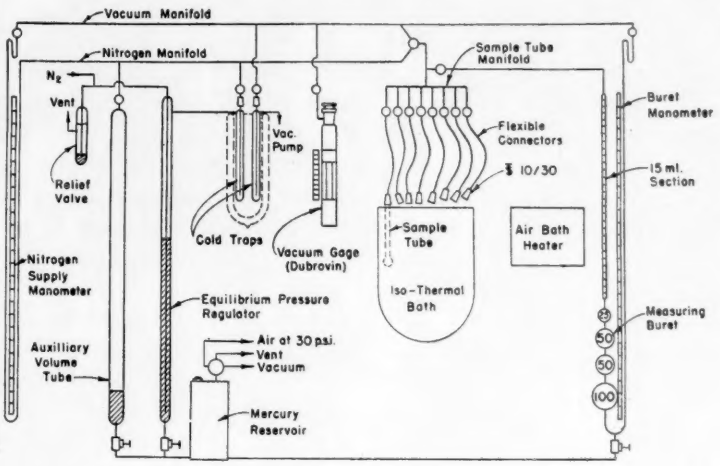
Captions of Figures 12-14

Fig. 12. In this design the manometer and burette are combined as a U-tube of 7-8 mm. i.d. tubing. Gregg and Sing[42] inserted a stopcock at F leading to the vacuum manifold and gas storage bulbs thereby overcoming the objection to degassing through the burette manometer tubing.

Fig. 13. Adsorption apparatus used at higher temperatures. M, manometer; C, mercury cutoff; B, calibrated burette; S, sample; U, ampoule breaker system; Y and X, upper and lower zero settings for manometer; L, entrance tube for helium and nitrogen, sealed off before breaking ampoule. Connections to vacuum and atmosphere were made through the large three-way stopcock on the manometer. The temperature in U is used to regulate the pressure in the burette B. Increments of gases are measured in the burette with the cut-off level at Y and are dosed into S by lowering the mercury to X. Further readings are made by raising the mercury in B.

Fig. 14. A robust and readily constructed apparatus.

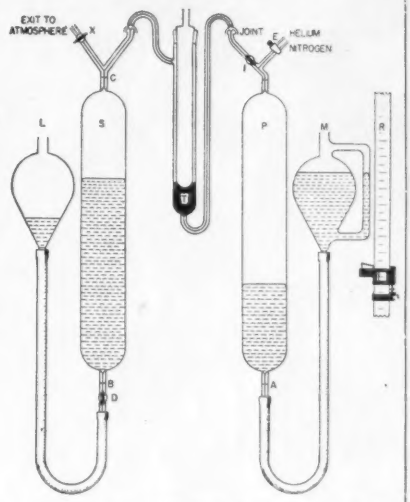
The Shell Group of Oil Companies [46]



by courtesy of Shell Development Co.

Fig. 15

W. V. Loebenstein and V. R. Deitz [47]



by courtesy of National Bureau of Standards

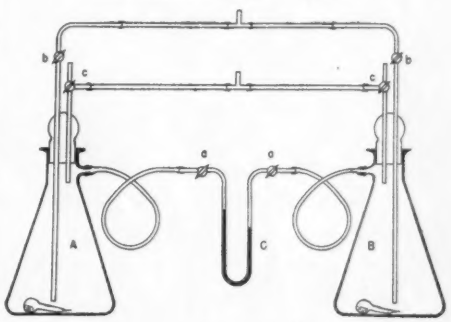
Fig. 16

Captions of Figures 15-16

Fig. 15. A number of sample bulbs are connected by separate stopcocks, flexible Tygon tubing and standard joints to the manifold. The samples are degassed, cooled, and allowed to adsorb gas at a constant pressure with all the stopcocks open until they have all reached equilibrium. All sample line stopcocks are then closed, the manifold is exhausted and then opened to the measuring burette. Each bulb, in turn, is then heated and the volume of gas desorbed from it is measured.

Fig. 16. Apparatus for the adsorption of nitrogen from helium mixtures at atmospheric pressure. S and P are a pair of 200 ml. cylindrical holders operated by mercury lifts L which are used to pass a mixture of helium and nitrogen back and forth through the sample. The total pressure of the mixture is read on the side-limb reservoir M, and a change in pressure due to the physical adsorption of the nitrogen in the mixture, is observed. The volume adsorbed is calculated from the known initial composition of the mixture, the volume of the holders, and the change in pressure. More than one point can be obtained by varying the composition of the mixture and repeating the process.

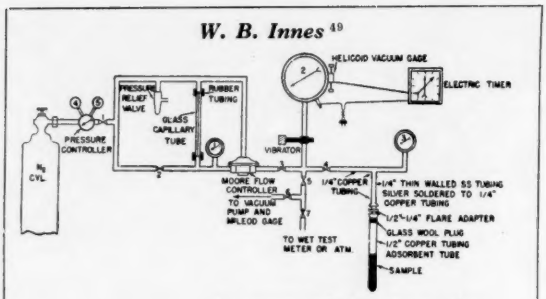
D. Smith and H. Green [48]



by courtesy of Industr. Engng. Chem.

Fig. 17

Fig. 17. A sealed ampoule containing a weighed amount of sample is placed in one of the flasks A and B filled with air humidified at a known R.H. The ampoule is then broken by shaking the flask, and the reading of the dibutylphthalate differential manometer C is taken as a measure of the surface area of the sample. The apparatus is capable of giving very good results in some cases.



by courtesy of Industr. Engng. Chem.

Fig. 18. Apparatus for continuous adsorption, surface area, and pore volume measurement.

attention to details of filling, sample preparation, etc. are certainly important and a rigidly standardised procedure must be adhered to.

Dynamic Methods

The apparatus shown in Fig. 18 was developed by Innes[49] and is a robust all-metal assembly designed around a Helicoid Vacuum Gauge. The method involves a slow flow of gas into the adsorbent system so that adsorption equilibrium is maintained as the pressure increases. Flow controllers and pressure regulators are used to ensure that the flow-rate is constant despite the gradual increase of (equilibrium) pressure in the adsorbent system. Adjustable contact points on the Helicoid gauge are arranged to operate a stopclock so that the time taken to reach an arbitrarily chosen pressure is automatically indicated. This duration is taken as being proportional to the surface area. The apparatus is robust and—once adjusted—easy to operate, but is subject to the usual limitations of single-point determinations.

Arnold[50] used the straight forward flow system shown in Fig. 19 for studying the adsorption of N_2/O_2 mixtures and included the analyser M for that purpose, but gas mixtures are probably better handled in glass circulating systems similar to that of White and Schneider[51]. Only one sample bulb is shown in the figure, but it should be possible to insert several and desorb separately as in the Shell apparatus.

A very useful approximate method for estimating the surface area, or more strictly the adsorption capacity, of a powdered or porous substance was first employed in 1934 by Pidgeon[52]. In this method, humidified air is passed at a constant rate through a bed of adsorbent until water vapour can be detected in the outlet by the change in electrical conductivity of a fused bead of calcium chloride. The passage of air at 25% R.H. for five seconds causes the resistance of the bead to fall from 10,000 to 500 ohm.

La Lande[53] uses the term 'Dry Gas Capacity' (D.G.C.) to denote the percentage by weight, of water, retained from a stream of humidified air by a bed of adsorbent, up to the point that water vapour appears in the exit gas. This point is found by the rather tedious procedure of repeatedly weighing a series of drying tubes connected to the gas outlet. An excellent straight-line relationship is obtained by plotting the D.G.C. results against the surface areas determined by Krieger's method. All points fall very closely on one or the other of two straight lines which appear to relate to either activation or sintering during pretreatment. The method seems to merit further attention and could easily be developed into a routine procedure if a means of automatically indicating the break-through point were to be incorporated[87].

Kinetic and Rate-of-Flow Methods

A number of attempts have been made to devise methods for the determination of specific surface based on the kinetics of gas adsorption and on the rate of gas flow through porous media. So far, these methods have all proved intractable either by reason of the experimental difficulties encountered in the observation of the desired data, or because the calculations involved are too complicated. Nevertheless, it should be possible to utilise these methods by adopting graphical and tabular solutions of the equations, fitted with constants obtained from preliminary observations. A detailed discussion of these methods and their applications is outside the scope of this review; the publications[54,55,56,57] should be consulted for further information.

EXPERIMENTAL TECHNIQUES—GRAVIMETRIC

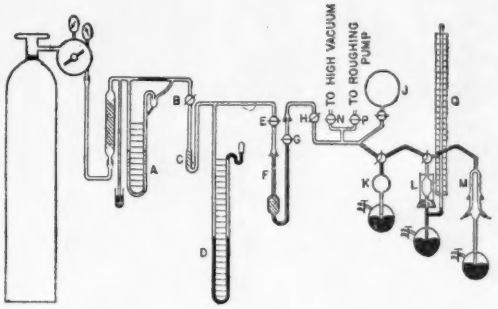
The gravimetric technique has the great advantage

that the volume of the adsorption system is quite immaterial, and the amount of gas adsorbed can be observed directly. This elimination of the tedious volume calibrations and dead space determinations, greatly simplifies the apparatus and the experimental technique. Furthermore, a single weight calibration, and buoyancy correction table will usually suffice for a very large number of runs.

The main disadvantage of the method, which has so far prevented it from completely ousting the volumetric techniques, is that the apparatus is much less robust and correspondingly more difficult to construct and maintain than volumetric apparatus. In addition, the apparatus has to be calibrated by placing known weights in the adsorbent-pan, and the method is consequently subject to the suspicion, always attached to determinations which are dependent on the constancy of the calibration, of easily fatigued or strained mechanical systems. Much of this objection has now been removed by the use of robust, commercially produced springs and by improved glass-working techniques.

Vol. 3 1953

J. R. Arnold[50]



by courtesy of J. Amer. Chem. Soc.

Fig. 19. The gas is passed through the regulating valve capillary flow manometer A and needle valves B and H at a known steady pressure reading on manometer D. After equilibrium has been reached the sample bulb F is shut off from the flow system and the gas desorbed into the measuring system K, J, L and Q.

Single Spring Balances

The original design of McBain and Bakr[58] is adequate for many purposes, and their paper gives a clear account of the minor difficulties, likely to be encountered in its use. Fig. 20 shows a typical modern apparatus[59] with a sensitivity of between 0.15 and 0.25mm. per microgram.

Boyd and Livingston[60] used a similar apparatus employing mercury cut-offs in the vapour handling and compressing system, but with a stopcock for dosing vapour into the balance case. The pressure was controlled by compressing the vapour in the dosing bulb, and read on a mercury manometer, oil manometer or McLeod gauge, according to the required pressure range.

Pidgeon described his own experimental techniques in considerable detail in the first[61] of his two major papers on hysteresis and water sorption. Adsorption and desorption points were taken at constant pressure by using as the adsorbate source a tube, containing adsorbate vapour in equilibrium with its liquid. The paper was mainly concerned with the origin of errors in the determination of hysteresis loops and Pidgeon's conclusion that 'the failure of other workers to observe true hysteresis loops was due to non-equilibrium pressure conditions during the admittance or withdrawal of vapour', was largely based on a a comparison of gravimetric and volumetric techniques. In his second paper[52] a modified apparatus is described for the study of the vapours of organic liquids which were introduced, by means of a hook pipette, into the trough leg of a mercury manometer, the other leg of which was connected to the manifold.

Seborg, Simmons and Baird[62] describe a simple arrangement for water sorption (Fig. 21) in which the sample is dried in a current of dry air, and the adsorption points are subsequently obtained by passing the air through saturators filled with solutions of known vapour pressure. This arrangement is typical of many systems used for routine testing.

Multiple Spring Balances

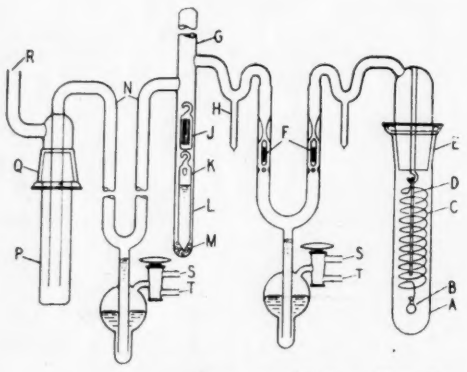
Another advantage of the gravimetric spring balance method is that several determinations can be carried out simultaneously by connecting the balance

cases to the same vapour manifold, and observing the individual spring extensions.

Seborg and Stamm[63] connected five or six simple spring units and humidifying bubblers in series, and immersed the whole assembly in a water bath. Condensation in the connecting tubes was prevented by heating them with a row of electric light bulbs.

The apparatus designed for the same purpose by Pidgeon and Maass[64] is more versatile and only slightly more complicated. Several spirals and an oil-filled U-tube manometer were immersed in the same bath, and vapour was admitted to the common manifold, by momentarily opening a tap leading to a liquid adsorbate reservoir kept at room temperature. The samples weighed about 0.1 g, the weight adsorbed was usually between 0.02 and 0.03 g, and the spring sensitivity was 0.5 mm. per mg., so that a total extension of about 1.25 cm. was usually observed.

F. E. Bartell and D. G. Dobay [59]



by courtesy of J. Amer. Chem. Soc.

Fig. 20. The spring C is mounted in a large tube A connected to the mercury float valves F, adsorbate ampoule K, breaker assembly J, M cut-off N and cold trap P. R leads to the high vacuum system used in degassing the sample. The adsorbate pressure is controlled by varying the temperatures of L and A. The glass rod D hung from the spring support serves as a datum for checking the alignment and level of the catheto-meter.

An apparatus for carrying out as many as fifteen simultaneous determinations is described by Milligan and his co-workers[65]. A fused silica spring is attached to a large ground glass sphere which seats in a spherical socket joint at one end of a closed glass tube, 1 metre long. Fifteen of these balance units are arranged linearly in a bath, the temperature of which can be controlled between −20 and +40°C. The vapour source is situated outside the bath and the vapour pressure is read on a differential oil manometer in the same plane as the adsorption bulbs. The spring depressions and manometer levels are observed with a vertically reading micrometer microscope mounted on a platform which traverses the length of the bath on a screwed rack. Adsorption and desorption points at high relative pressures are made at nearly constant pressure by adding a 20 l. ballast volume to the adsorption manifold. A high-speed oil pump with low-temperature vapour trap, phosphorus pentoxide trap and by-pass valves is needed to degas this system adequately. In the opinion of the writer the constant pressure conditions are much more readily obtained by Pidgeon's method[61]. Milligan gives a useful discussion of the practical problems connected with the choice of construction materials, stopcock grease, etc. He recommends the use of ' dead soft ' dental platinum foil 0.0005 inch thick for fabricating the adsorption buckets.

The apparatus cannot be easily modified for use with very low-temperature adsorbates such as nitrogen, but no difficulty should be experienced in constructing a glass-fronted refrigeration chamber for the adsorption of CF_2Cl_2 at −30 to −50°C.

Stamm and Woodruff[66] mount six balance tubes, vapour source and manometer on a turntable and immerse the whole assembly in a large bath of aqueous glycol solution, the temperature of which is controllable between −20 and +50°C. The liquid adsorbate in the vapour source bulb is agitated by a non-splash stirrer, and its temperature is controlled by an electric heater and a cooling coil situated together with the bulb and a thermometer in a small Dewar flask in the main glycol bath.

Each spring can be read in turn by rotating the turntable so that both the spring extension and the

manometer reading can be read together on a cathetometer located outside the bath. The apparatus is mainly used for routine testing of the water adsorptive capacity of wood and cellulose, but it can be used generally for studying the adsorption of any liquid which has a vapour pressure of about 10 cm. Hg at temperatures between −20 and +50°C. The sensitivity of the short springs is not high enough to allow for the observation of adsorptions at low relative pressures.

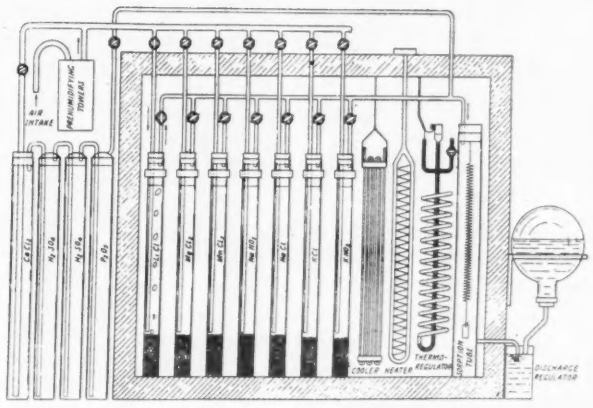
High Pressure Sorption Spring Balances

The gravimetric method is practically the only means of determining adsorption isotherms at high absolute pressures. Two outstanding examples of this technique are mentioned below.

Morris and Maass[67] used a high pressure gravimetric balance for the study of adsorption effects in the critical pressure regions. It consisted of an inverted U-tube of thick-walled Pyrex glass, with the spring and bucket containing the adsorbent in one limb and liquid adsorbent in the other.

Khodot[68] has used this technique for the study of the adsorption of methane by coal at high pressures, similar to those which exist in the coal seams. He used a quartz spring balance suspended in a transparent plastic tube bonded and strapped to a steel housing fitted with steel capillary lines leading to needle valves and a dead-weight pressure gauge. The free volume and buoyancy corrections were calculated from the results of blank runs with hollow glass spheres adjusted to have the same density as the coal under investigation. Satisfactory sorption isotherms were obtained at pressures up to 50 atm.

C. O. Seborg, F. A. Simmonds and P. K. Baird[62]



by courtesy of Industr. Engng. Chem.

Fig. 21. A single water sorption gravimetric apparatus. Dry air is humidified by passing it through one of the saturators containing $MgCl_2$. LiCl, etc. The humidified air is passed through the sorption tube and the depression of the spring noted for each humidity value.

Beam Balances

There are two types of beam balances suitable for adsorption work, depending on whether extremely high sensitivity at very low total loads or medium sensitivity at large total loads is required. These qualities should be contrasted with those of the normal short spring balance which has a medium sensitivity at low total loads. The majority of high-sensitivity low-load balances are based on those originally designed by Barrett[69] and by Gulbransen[70].

Barrett, Birnie and Cohen[69] used a glass beam about 40 cm. long supported on a tungsten torsion wire and enclosed the whole assembly in a tubular glass casing connected to the vapour and vacuum manifolds. Calibration was effected by moving a small soft-iron rider along the beam, by means of a magnet outside the balance case. The sensitivity was about one-tenth of a microgram at a total load of 0.5 g.

Gulbransen's balance[70] is constructed from glass rod, quartz fibres and metal wire, on the same principles as an ordinary chemical beam balance.

Rhodin[71] modified this apparatus by using thinner

and lighter wires to increase the sensitivity of the balance to better than one-tenth of a microgram at a load of 0.5 g. The construction of this type of balance demands great attention to detail and much manipulative skill so that the information given in the original papers is not adequate in all respects.*

Quartz and glass micro-balances are reviewed by El-Badry and Wilson[72] who also give full constructional details of their own balance. This balance could be used for adsorption studies by assembling it in a tubular housing, but it should be emphasised that quartz torsion balances are not at all easy to build.

Gregg[73] has designed an adsorption balance which is much simpler to construct than would appear from the figures in his paper. It has a sensitivity of about 300 microgram, independent of the total load which may be as large as 20 g. The ease of construction and large load-bearing capacity are due to the fact that the balance beam is a rigid assembly of Pyrex rod and tubing. Balancing is effected by varying the current in a solenoid, external to the balance case, which interacts with another solenoid suspended from the counterpoise balance arm inside the case. A later apparatus, not yet described in the literature, uses a low-hysteresis ' Alnico ' magnet instead of the solenoid. This eliminates the rather troublesome spring-leads inside the balance case, without re-introducing magnetic hysteresis effects. The beam of the new apparatus is a 26 cm. length of 5 mm. Pyrex rod resting on a removable carriage. The same design has been further developed to provide for automatic balancing and recording.[74]

MISCELLANEOUS TECHNIQUES

Two techniques, which cannot be classified under any of the previous headings have been devised by Chambers and King and by Pierce and Smith.

Chambers and King[75] use Pyrex glass tubing and and tungsten rods to build a Nicholson type hydrometer, which floats on mercury in an adsorption chamber. The float carries a sample load of 6 g., and the minimum observable displacement (0.02 mm.) corresponds to a change in load of 0.3 mg. This represents a (minimum displacement weight) : (load) ratio of 1 : 20,000 which compares favourably with the usual spring balance ratio of 1 : 7,000. The sample can be degassed by lowering the mercury out of the chamber, and heating and evacuating in the normal manner.

Pierce and R. N. Smith[76] adopted the unusual procedure of directly weighing the sample and adsorbate in a detachable bulb and stopcock assembly. The method is rather slow, since the bulb has to be taken off, dried, weighed, and replaced for every adsorption point, but this disadvantage is offset by the elimination of volume and dead space calculations. It is probably not satisfactory for the observation of adsorption-desorption hysteresis, owing to the uneven local temperature and pressure distribution in the bulb during weighing.

SPECIAL TOPICS

There are a number of noteworthy papers covering various specific aspects and applications of adsorption techniques. Some of these are quoted below under their respective headings.

Comparison of Methods

An excellent, but short, account of the theoretical basis of the gas adsorption method and the experimental techniques of adsorption, heat of wetting, and dyestuff adsorption methods of determining surface areas has been given by Gregg[77].

Similar accounts, but with more emphasis on the direct comparison of experimental results have been published by Ewing[78] covering dyestuff adsorption, and Maggs[79] covering heat of wetting methods, whilst Emmett and De Witt[80] report on the use of very large numbers of adsorbents to compare the specific surfaces obtained by fatty acid and radioactive salt adsorption, ultramicroscope counting and gas adsorption methods.

The Cross Sectional Areas of Adsorbed Molecules

Two papers published by Livingston are generally

* See also—Bowers, R., *Phil. Mag.*, **44**, (1954), 467. ' The Adsorption of Gases at High Saturation.'

Vol
3
195

considered the standard source for cross sectional area data. Unfortunately most of Livingston's figures are based on a comparison of monolayer volumes taking the standard C.A. of nitrogen as 16.2 $Å^2$ whereas modern work favours 15.8 $Å^2$. Any adsorbates not listed in these papers can be found in Harris's review of adsorbates and adsorbents[83].

Pore-Size Computations

The two principle methods used for the computation of pore size distributions from desorption data are those of Barrett[84] and Pierce [85]. Pierce's method is the simpler of the two but not so well founded theoretically. Barrett's method should be used when the majority of the pores have radii less than about 40 Å.

Barrett and Joyner[86] draw attention to the necessity of adsorbing a considerable excess of gas at saturation before commencing desorption, and to the errors caused by using a faulty vapour pressure thermometer for the observation of P_o.

CONCLUSIONS

More than 300 papers on the experimental techniques of gas adsorption have been published during the last 15 years, but the 56 different types of apparatus referred to in the present review should be sufficiently representative to enable a method suitable for any given purpose to be selected.

The choice can be narrowed even further by following the current trend to use special apparatus only when it is likely to be in use for a considerable time, or when some unique feature is absolutely essential. Otherwise it is now customary to find only one of three principle types of assembly in any one laboratory, depending mainly on the function of the laboratory.

University or Research Establishment laboratories engaged in surface studies usually have a Tompkins and Young apparatus available for general use, whilst industrial laboratories frequently have both, a conventional Emmett system for standardisation and research purposes, and a rapid or multi-sample surface area tester for batch control and product ' analysis '.

In recent years, a further trend towards the use of the Gregg sorption balance is evident in many laboratories, even in the works laboratories. This is because the balance can be used for other purposes, such as differential thermal analysis, decomposition temperature determination, reactivity and stability tests under various conditions, and even for magnetic susceptibility studies. This versatility, taken in conjunction with the continuous recording facilities, outweighs the greater difficulty of construction. It may be predicted that within a few years the Gregg and the Tompkins and Young apparatus will share the field between them.

This paper is published by the permission of Dr. A. Parker, Director of Fuel Research, Department of Scientific and Industrial Research.

References

In order to facilitate tracing of the information quoted by subject the numbers of the references set out below have been arranged in tabulated form, see Table Nos. I and II below.

All methods are divided into two major groups in accordance with the technique employed :—

I. The Volumetric Method
II. The Gravimetric Method

Further, all methods are divided into 2 sections indicating their uses as follows :—

(*a*) Suitable for Routine Work
(*b*) Suitable for Research Work

Finally, sub-headings have been introduced, indicating characteristics of apparatus design or special features of the procedures described.

It should be noted that research apparatus suitable for, or readily adapted to, routine purposes have been listed under (*a*) *and* (*b*); most of the remaining methods shown under (*a*) are simplified to an extent which makes them unsuitable for accurate research work.

The list of References is not exhaustive. Only the most important of the papers published before 1935 have been quoted. Earlier References can be found readily in the bibliographies appended to the publications quoted against Nos. 4, 5, 6 and 7.

I. VOLUMETRIC METHODS

Technical Features

Type	*Reference Nos.*
Conventional Apparatus	8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19
Modified Conventional Apparatus	34, 35, 36, 37, 38, 39, 40, 41, 42, 43
Measurement of Small Adsorptions	20, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32, 33
Dynamic Methods	49, 50, 51, 52, 53
Kinetic Methods	54, 55, 56, 57
Miscellaneous Designs and Methods	44, 45, 46, 47, 48

Applications

Suitable for:	*Reference Nos.*
(*a*) Routine Work	14, 17, 19, 22, 31, 34, 37, 38, 41, 42, 45, 46, 47, 48, 49, 50, 52, 53,
(*b*) Research Work	9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 20, 21, 22, 23, 24, 25, 26, 28, 29, 30, 32, 33, 39, 40, 41, 42, 44, 45, 50, 51

II. GRAVIMETRIC METHODS

Technical Features

Type	*Reference Nos.*
Single-Spring Balance	52, 58, 59, 60, 61, 62
Multiple-Spring Balance	63, 64, 65, 66
High-Pressure Sorption Spring Balance	67, 68
Beam Balance	69, 70, 71, 72
Miscellaneous Designs	75, 76

Applications

Suitable for:	*Reference Nos.*
(*a*) Routine Work	52, 53, 61, 62, 64, 65, 66
(*b*) Research Work	58, 59, 60, 61, 67, 68, 69, 70, 71, 72, 73, 74, 75

1 EMMETT, P. H., BRUNAUER, S. and TELLER, W. E., *J. Amer. Chem. Soc.*, **60,** (1938), 309. 'Adsorption of Gases in Multimolecular Layers'.

2 HILL, T. L., *Advances in Catalysis* (Academic Press Inc., New York, 1952), IV.

3 TOMPKINS, F. C., *Ann. Rep. Prog. Chem.*, **47,** (1950), 54. 'Adsorption and Surface Chemistry'.

4 ELEY, D. D., *Ann. Rep. Progr. Chem.*, **49,** (1952), 18. 'Surface Chemistry'.

5 BRUNAUER, S., *The Adsorption of Gases and Vapours*, (Oxford University Press, London, 1944).

6 MCBAIN, J. W., *The Sorption of Gases by Solids*, (George Routledge & Sons Ltd., London, 1952).

7 GREGG, S. J., *The Surface Chemistry of Solids*, (Chapman & Hall Ltd., London, 1951).

8 EMMETT, P. H. and BRUNAUER, S., *J. Amer. Chem. Soc.*, **56,** (1934), 35. 'The Adsorption of Nitrogen by Iron Synthetic Ammonia Catalysts'.

9 EMMETT, P. H. and BRUNAUER, S., *J. Amer. Chem. Soc.*, **59,** (1937), 310. 'Accumulation of Alkali Promoters on Surfaces of Iron Synthetic Ammonia Catalysts'.

10 EMMETT, P. H. and BRUNAUER, S., *Trans. Electrochem. Soc.*, **71,** (1937), 383.
'The Use of Adsorption Isotherms for Measuring the Surface Area of Catalysts and Other Finely Divided Materials'.

11 EMMETT, P. H., *12th Report of the Committee on Catalysts*, (John Wiley & Sons Inc., New York, 1940) Chapter IV.
'Physical Adsorption in the Study of the Catalysts Surface".

12 EMMETT, P. H., *Advances in Colloid Science*, (Interscience Publications Inc., New York, 1942), I.
"The Measurement of the Surface Areas of Finely Divided or Porous Solids by Low Temperature Adsorption Isotherms.

13 EMMETT, P. H., *A.S.T.M. Special Tech. Publ.* No. **41,** (1951), 95.
'A New Method for the Determination of Surface Area'.

14 JOYNER, L. G., *Scientific and Industrial Glass Blowing and Laboratory Techniques*, edited by W. E. Barr and V. J. Anhorn, (Instruments Publishing Co., Pittsburgh, 1949), 257.
'Gas Adsorption Apparatus for Measuring Surface Areas'.

15 BARR, W. E. and ANHORN, V. J., *Instruments*, **20,** (1947), 454.
'Scientific Glassblowing and Laboratory Techniques, XII. Gas Adsorption Apparatus for Measuring Surface Areas'.

16 HARKINS, W. D. and JURA, G., *J. Amer. Chem. Soc.*, **66,** (1944), 1366.
'Method for the Determination of the Area of a Solid without the Assumption of a Molecular Area, and the Areas Occupied by Nitrogen and other Molecules on the Surface of a Solid'.

17 RIES, H. E., NORDSTRAND, R. A. VAN, and TETER, J. W., *Industr. Engng. Chem.*, (*Industr.*), **37,** (1945), 310.
'Surface Area of Catalysts'.

18 SMITH, W. R., THORNHILL, F. S. and BRAY, R. I., *Industr. Engng. Chem.*, (*Industr.*), **33,** (1941), 1303.
'Surface Area and Properties of Carbon Black'.

19 HAERTLEIN, J. B. and SACHSE, J. F., *A.S.T.M. Special Tech. Publ.* No. **140,** (1952).
Symposium on Testing Metal Powders. 'Some Experience in Specific Surface Measurement of Metal Powders by Low Temperature Gas Adsorption'.

20 CRAWFORD, V. A. and TOMPKINS, F. C., *Trans. Faraday Soc.*, **44,** (1948), 698.
'The Adsorption of Gases. SO_2, NH_3, CO and N_2O on BaF_2 Crystals'.

21 TOMPKINS, F. C. and YOUNG, D. M., *Trans. Faraday Soc.*, **47,** (1951), 77.
'The Adsorption of Gases on Caesium Iodide'.

22 CROWELL, A. D. and YOUNG, D. M., *Trans. Faraday Soc.*, **49,** (1953), 1080.
'The Adsorption of Argon on Graphite'.

23 YOUNG, D. M., *Rev. Sci. Instrum.*, **24,** (1953), 77.
'A Doser for Admitting Measured Amounts of Vapour'.

24 ORR, W. J. C., *Proc. Roy. Soc.*, **173A,** (1939), 349.
'The Adsorption of Non-Polar Gases on Alkali-Halide Crystals'.

25 RIDEAL, E. K. and TRAPHELL, B. M. W., *Proc. Roy. Soc.*, **205A,** (1951), 409.
'Oxygen and Carbon Monoxide Chemisorption on Evaporated Tungsten Films'.

26 WOOTEN, L. A. and BROWN, C. *J. Amer. Chem. Soc.*, **65,** (1945), 113.
'Surface Areas of Oxide-Coated Cathodes by Gas Adsorption'.

27 BURWELL, R. L., SMUDSKI, P. A. and MAY, T. P., *J. Amer. Chem. Soc.*, **69,** (1947), 1525.
'Ethylene Adsorption Isotherms at −183°C'.

28 ARMBRUSTER, M. H. and AUSTIN, J. B., *J. Amer. Chem. Soc.*, **60,** (1938), 467.
'The Adsorption of Gases on Plane Surfaces of Mica'.

29 ARMBRUSTER, M. H. and AUSTIN, J. B., *J. Amer. Chem. Soc.*, **61,** (1939), 1117.
'The Adsorption of Ethyl Iodide on a Plane Surface of Iron at 20°C'.

30 GANS, P. M., BROOKS, U. S. and BOYD, G. E., *Industr. Engng. Chem.*, (*Anal.*), **14,** (1942), 396.
'Adsorption of Vapours by Crystalline Solid Surfaces'.

31 LADISCH, R. K., MCQUE, B. and KNESBACH, S. L., *Analyt. Chem.*, **26,** (1954), 399.
'Method of Measuring Ultra-Fine Glass-Fibres'.

32 JURA, G. and HARKINS, W. D., *J. Amer. Chem. Soc.*, **66,** (1944), 1356.
'Determination of the Decrease of Free Surface Energy of a Solid by an Adsorbed Film'.

33 BANGHAM, D. H. and MOSALLAM, S., *Proc. Roy. Soc.*, **165A,** (1938), 552.
'The Adsorption of Vapours at Plane Surfaces of Mica'.

34 BUGGE, P. E. and KERLOGUE, R. H., *J. Soc. Chem. Ind.* (*London*), **66,** (1947), 377.
'The Determination of the Surface Area of Powders by Means of Low-Temperature Adsorption Isotherms'.

35 BUGGE, P. E., KERLOGUE, R. H. and WESTWICK, F., *Nature*, **158,** (1946), 28.
'Surface Area Determination'.

36 SALVI, G. and FIUMARA, A., *Riv. Combust*, **6,** (1953), 345.
'L'Adsorbimento Nella Misura Delle Area Superficiali di Corpo Porosi'.

37 SMITH, D. E., *The Petrol. Engr.*, **24,** (1952), C–52.
'A Simplified Surface Area Apparatus and Catalyst Tester'.

38 DEITZ, V. R. and GLEYSTEEN, L. F., *J. Res. Nat. Bur. Stand.*, **29,** (1942), 191.
'Surface Available to Nitrogen on Bone Black and Other Carbonaceous Adsorbents'.

39 *Fuel Research*, (H.M.S.O., London, 1952), 1951, **25.**

40 JOYNER, L. G., WEINBERGER, E. B. and MONTGOMERY, C. W., *J. Amer. Chem. Soc.*, **67,** (1945), 2182.
'Surface Area Measurements of Activated Carbons, Silica Gel and Other Adsorbents'.

41 KRIEGER, K. A., *Industr. Engng. Chem.*, (*Anal.*), **16**, (1944), 398.
'Apparatus for Surface Area Measurements'.

42 GREGG, S. J. and SING, K. S. W., *J. Phys. Chem.*, **55**, (1951), 592.
'The Effect of Heat Treatment on the Surface Properties of Gibbsite'.

43 ANDERSON, R. B., *Industr. Engng. Chem.*, (*Anal.*), **18**, (1946), 156.
'Improved Adsorption Vessel'.

44 HARRIS, B. L. and EMMETT, P. H., *J. Phys. Chem.*, **53**, (1949), 811.
'Adsorption Studies'.

45 ASKEY, P. J. and FEACHEM, C. G. P., *J. Soc. Chem. Ind.*, **57**, (1938), 272.
'The Comparison of the Specific Surface of Finely-Divided Solids'.

46 The Royal Dutch Shell Group of Oil Companies, *Shell Method Series No.* 666/49U.
'Determination of Surface Properties of Porous Solids—Low Temperature Nitrogen Adsorption Method'.

47 LOEBENSTEIN, W. V. and DEITZ, V. R., *J. Res. Nat. Bur. Stand.*, **46**, (1951), 51.
'Surface Area Determination by Adsorption of Nitrogen from Nitrogen-Helium Mixtures'.

48 SMITH, D. and GREEN, H., *Industr. Engng. Chem.*, (*Anal.*), **14**, (1942), 382.
'Measuring the Specific Surface of Particulate Substances—A Water Vapour Adsorption Method'.

49 INNES, W. B., *Analyt. Chem.*, **23**, (1951), 759.
'Apparatus and Procedure for Rapid Automatic Adsorption, Surface Area and Pore Volume Measurement'.

50 ARNOLD, J. R., *J. Amer., Chem. Soc.*, **71**, (1949), 104.
'Adsorption of Gas Mixtures'.

51 WHITE, L. and SCHNEIDER, C. H., *J. Amer. Chem. Soc.*, **71**, (1949), 2593.
'Physical Adsorption from Mixtures of Gases'.

52 PIDGEON, L. M., *Canad. J. Res.*, **12B**, (1935), 41.
'The Sorption of Vapours by Active Silica'.

53 LA LANDE, W. A., MCCARTER, W. S. M. and SANBORN, J. B., *Industr. Engng. Chem.*, (*Industr.*), **36**, (1944), 99.
'Bauxite as a Drying Adsorbent'.

54 SUTHERLAND, K. L. and WINFIELD, M. E., *Austral. J. Chem.*, **6**, (1953), 221.
'Transient Rates of Gas Sorption'.

55 JURA, G. and POWELL, R. E., *J. Chem. Phys.*, **19**, (1951), 251.
'Kinetics of Gas Adsorption as a Method of Area Determination'.

BLOCKER, H. G., CRAIG, S. L. and ORR, C., *J. Phys. Chem.*, **57**, (1953), 517.
'Dynamic Gas Adsorption Methods of Surface Area Determination'.

57 BARRER, R. M., *Brit. J. Appl. Phys.*, *Suppl.* 3, (1954), 41.
'The Measurement of Surface Area of Finely-Divided Solids by the Flow of Gas through Them'.

58 MCBAIN, J. W. and BAKR, A. M., *J. Amer. Chem. Soc.*, **48**, (1926), 690.
'A New Sorption Balance'.

59 BARTELL, F. E. and DOBAY, D. G., *J. Amer. Chem. Soc.*, **72**, (1950), 4388.
'The Adsorption of Aliphatic Amine Vapours on Silica Gel'.

60 BOYD, G. E. and LIVINGSTON, H. K., *J. Amer. Chem. Soc.*, **64**, (1942), 2838.
'Adsorption and the Energy Changes of Crystalline Solid Surfaces'.

61 PIDGEON, L. M., *Canad. J. Res.*, B.10, (1934), 713.
'Hysteresis in Silica Gel Sorption Systems'.

62 SEBORG, C. O., SIMMONDS, F. A. and BAIRD, P. K., *Industr. Engng. Chem.*, (*Industr.*), **28**, (1936), 1245.
'Sorption of Water Vapour by Paper-Making Materials'.

63 SEBORG, C. O. and STAMM, A. J., *Industr. Engng. Chem.*, (*Industr.*), **23**, (1931), 1271.
'Sorption of Water Vapour by Paper-Making Materials'.

64 PIDGEON, L. M. and MAASS, O., *J. Amer. Chem. Soc.*, **52**, (1930), 1053.
'The Adsorption of Water by Wood'.

65 MILLIGAN, W. O., SIMPSON, W. C., BUSHEY, G. L., ROCHFORD, H. H. and DRAPER, A. L., *Analyt. Chem.*, **23**, (1951), 739.
'Precision, Multiple, Sorption - Desorption Apparatus'.

66 STAMM, A. J. and WOODRUFF, S. A., *Industr. Engng. Chem.*, (*Anal.*), **13**, (1941), 386.
'A Convenient Six - Tube Vapour Sorption Apparatus'.

67 MORRIS, H. E. and MAASS, O., *Canad. J. Res.*, 9.*B*, (1933), 240.
'Discontinuities in the Adsorption of Gases, Vapours and Liquids on Solid Surfaces at the Critical Temperature under Critical Pressure'.

68 KHODOT, V. V. and YANOVSKAYA, M. F., *Dokl. Akad. Nauk S.S.S.R.*, **97**, (5), (1954), 879.
'Use of a Quartz Sorption Balance for Investigation of the Sorption Capacity of Coals at High Pressures'.

69 BARRETT, H. M., BIRNIE, A. W. and COHEN, M., *J. Amer. Chem. Soc.*, **62**, (1940), 2839.
'The Adsorption of Water Vapour on Silica Surfaces by Direct Weighing'.

70 GULBRANSEN, E. A., *Rev. Sci. Instrum.*, **15**, (1944), 201.
'A Vacuum Micro-Balance for the Study of Chemical Reactions on Metals'.

71 RHODIN, T. N., *J. Amer. Chem. Soc.*, **72**, (1950), 4343.
'Studies of Copper Surfaces by Low Temperature Adsorption Isotherms'.

72 EL-BADRY, H. M. and WILSON, C. L., *Roy. Inst. Chem. Monograph*, **No. 4**, (1950), 23.
'The Construction and Use of a Quartz Microgram Balance'.

73 GREGG, S. J., *J. Chem. Soc.*, (1946), 561, 564.
'An Electrical Sorption Balance'.

74 GREGG, S. J. and WINTLE, M. F., *J. Sci. Instrum.*, **23**, (1946), 259.
'An Automatically Recording Electrical Sorption Balance'.

75 CHAMBERS, H. H. and KING, A., *J. Chem. Soc.* (1939), 139.
'A New Technique for the Measurement of Adsorption of Gases and Vapours'.

[76] PIERCE, C. and SMITH, R. N., *J. Phys. Chem.*, **52,** (1948), 1111.
'The Adsorption of Ethyl Chloride by Graphite'.

[77] GREGG, S. J., *Trans. I. Chem. E.*, **S.25,** (1947), 40.
'Adsorption and Heat of Wetting Methods of Measuring Surface Area'.

[78] EWING, W. E. and RHODA, R. N., *Analyt. Chem.*, **22** (1950), 1453.
'Determination of Relative Specific Surface of ZnO Pigments'.

[79] MAGGS, F. A. P., *B.C.U.R.A. Bull.*, **9,** (1945), 253.
'Assessment of Specific Surface of Porous Solids by Adsorption Methods'.

[80] EMMETT, P. H. and WITT, T. DE, *Industr. Engng. Chem.*, (*Anal.*), **13,** (1941), 28.
'Determination of Surface Areas—Pigments, Carbon Blacks, Cement and Miscellaneous Finely Divided or Porous Materials'.

[81] LIVINGSTON, H. K., *J. Amer. Chem. Soc.*, **66,** (1944), 569.
'The Cross-Sectional Areas of Molecules Adsorbed on Solid Surfaces'.

[82] LIVINGSTON, H. K., *J. Colloid Sci.*, **4,** (1949), 447.
'Cross-Sectional Areas of Molecules Adsorbed on Solid Surfaces'.

[83] HARRIS, B. L., *Industr. Engng. Chem.* (*Industr.*), **46,** (1954), 64.
'Adsorption'.

[84] BARRETT, E. P., JOYNER, L. G. and HALENDA, P. P., *J. Amer. Chem. Soc.*, **73,** (1951), 373.
'The Determination of Pore Volume and Area Distribution in Porous Substances'.

[85] PIERCE, C., *J. Phys. Chem.*, **57,** (1953), 149.
'Computation of Pore Sizes from Physical Adsorption Data'.

[86] BARRETT, E. P. and JOYNER, L. G., *Analyt. Chem.*, **23,** (1951), 791.
'Determination of Nitrogen Adsorption/Desorption Isotherms'.

[87] ANSBACHER, F. and JASON, A. C., *Nature*, **171,** (1953), 177.
'Effect of Water Vapour on the Electrical Properties of Anodised Aluminium'.

Vol.
3
1953

Vacuum, Vol. III No. 3 *July 1953,*

FREEZE DRYING AND THE TISSUE BANK

H. H. G. Eastcott, M.S., F.R.C.S. *Assistant Director, Surgical Unit, St. Mary's Hospital Medical School, London, W.1.**

Summary

THE CLINICAL ASPECTS of tissue transplantation are discussed. Non-vital methods of tissue storage to facilitate transplantation are considered. After a short survey of atmospheric methods the author deals extensively with the merits of the freeze drying technique and gives details of a practical method of freeze drying arteries for the purpose.

Sommaire

DISCUSSION DES ASPECTS cliniques de la transplantation de tissus. L'on tient compte des méthodes de conservation *in vitro* de tissus, facilitant les transplantations. Après avoir rapidement passé en revue les méthodes atmosphériques, l'auteur traite particulièrement des méthodes de lyophilisation et donne des détails de méthodes pratiques de lyophilisation d'artères pour ce besoin.

Vol 3 195

INTRODUCTION

THE PROSPECT OF grafting tissues from one individual to another has always attracted surgeons. John Hunter was actively interested in this problem and conducted many experiments in the transplantation of tissues such as skin and teeth. It is now abundantly clear that seldom do such transplants survive for long in the body of the host animal. The reason for this, and the manner of the body's reaction to foreign cells, was long considered to be a local reaction, see Loeb[1], but Medawar[2] showed that in fact living cells, like other proteins, act as antigens. An immune response was shown to result from skin grafts in rabbits and the time of survival of successive crops of grafts from the same donor to the same recipient was found to decrease very considerably.

THE FATE OF TISSUE TRANSPLANTS

Only in highly inbred strains of laboratory animals or sometimes in identical twins is tissue transplantation followed by survival of the actual cells of the graft. An exception to this almost universal rule is the survival of cells from another individual, or even another species, when grafted into the anterior chamber of the eye. It may be that in the absence of vascular connections between the graft and its surroundings such grafts are virtually no more than a specialised kind of tissue culture preparation. Indeed it has often been noticed that such grafts undergo degeneration if newly formed blood vessels should reach them from the iris, for example. The vehicle for immunological exchanges is perhaps provided in this way.

In the writer's opinion, perhaps the only instance in clinical practice where tissues from another individual (homografts) are likely to survive, is corneal grafting. This may be so because the graft is placed over the anterior chamber. It is known that experimental corneal grafts which have been deliberately killed by unprotected freezing, for example, always become opaque with a dense fibrous change which must have been derived from the surrounding living

* MS. received June, 1954.

tissue.

In most other situations, however, homografts and heterografts undergo a regular sequence of changes as follows :—

(1) A transient phase of continued activity which may include actual growth[3]. This varies greatly with the tissue and species concerned. In some tissues such as bone, it has never been shown to exist.

(2) A local foreign body reaction, which is at its least in homografts.

(3) A slow process of substitution and replacement, sometimes with degenerative changes such as calcification.

It is generally believed that the extent and rapidity of these changes is directly proportional to the chemical difference between the graft and the recipient.

The overwhelming majority of grafts between other parts of different individuals undergo gradual degeneration but in the process their substance becomes replaced by what has been termed ' creeping substitution '.

In general, when such reactive phenomena are well marked, failure of the purpose for which the graft was used is to be expected; in much the same way as infection leads to its destruction or extrusion. Every effort is therefore made in the selection and preservation of tissues for grafting to use tissues, whose specificity and freshness differ as little as possible from those of the host.

THE STORAGE OF TISSUE

General

It is by no means certain that grafts must necessarily be alive at the time of implantation. Past experience suggested that they should, but probably only because living tissues are almost certain to be in perfect chemical preservation and therefore, in the ways already described, more suitable for use as grafts than dead tissues, indifferently preserved, whose protein might well have undergone denaturation since cellular death. But with the development of efficient techniques for storing biological material over long periods this risk has been greatly diminished.

Let us then consider the advantages of non-vital methods of storage. Firstly, they offer practical means of keeping proteins in a stable and un-denatured condition for indefinite periods. Secondly, in the case of tissues, since cells need not be kept alive, the opportunity may arise to kill the bacteria within them in various ways which would not be possible in life. Thirdly, there is good evidence that in some circumstances the normal reactions of a living graft may prejudice the result of the operation, as, for example, by causing clotting in blood vessel grafting, and in fact a dead graft may be free from this objection[4].

Present Methods for long-term tissue banking include:

- Chemical Fixation
- Deep Freezing
- Cathode and X-Irradiation
- Freeze Drying

The last forms the main subject of this paper.

Chemical Fixation

This method has the advantage of simplicity and of the disinfectant properties of the substance used. Protein is inevitably denatured and few grafts are suitable for preservation in this way. Bone and blood vessels can be banked in merthiolate and formalin respectively but the results do not equal those obtainable with un-denatured materials.

Deep Freezing

Rapid freezing followed by storage at low temperatures, as in the food industry, has proved to be a most satisfactory method for tissue banking. Though in most instances the graft is killed by this process, the results of using frozen bone and blood vessel grafts (Fig. 1) have been excellent[5,6,7]. Tissues frozen in 15% glycerol in Ringer saline solution have been shown to survive for many months, for example semen[8], blood[9], and cornea[10]. The principal objection to deep freezing is the need to maintain at all times the low temperatures (–20° to –79°C) which are necessary to prevent deterioration in the stored tissue. Another disadvantage is that most bacteria are able to withstand the freezing and thawing processes and can remain alive during the prolonged

Fig. 1 (*a*). Thrombosis of the popliteal artery, which caused severe limitation of the blood supply to the calf muscles, and crippling pain on walking more than 50 yards (see Fig. 1b.)

storage periods which deep freezing allows.

Cathode and X-Irradiation

Ionising radiations can now be used as a means of inactivating both the enzyme systems and the bacterial content of stored tissues. Denaturation sometimes accompanies this treatment but it has been suggested[11] that if the grafts are frozen at -79°C during the irradiation these changes are prevented. Food can be kept at room temperatures after such processing[12] but no report has yet been made of the results of grafting tissues kept above their freezing point after irradiation.

THE FREEZE DRYING TECHNIQUE

(a) General

World War II brought great advances in the treatment of war wounds, mostly due to the general availability, even in forward areas, of blood plasma and penicillin. These complex protein substances could only be stored and transported under service conditions after the problem of their production and preparation in bulk had been solved by the use of freeze drying.

The application of freeze drying to the needs of the tissue bank was inevitable; considerable fundamental research is now being undertaken in order to develop the method and to determine necessary techniques for producing the best possible grafts. Much has already been achieved: The transplantation of freeze-dried bone is now a routine method in some institutions. Dried donor skin has been successfully used as a temporary dressing in extensive burns pending autogenous skin grafting; in both instances large collections of material can be obtained by the use of cadaver donors[13].

Arterial grafts seem to be ideally adapted to this type of storage; they are scarce and the clinical demand for them is increasing. Up to the present most artery banks have employed the method of refrigeration in complex nutrient saline media; this necessitated the rejection of unused grafts after approximately a month. Deep freezing the grafts avoids this disadvantage, but, though storage is less complicated, transportation of the material is quite difficult; also there is always some risk of loss of the banked material from failure of the freezing device.

Of the methods which permit grafts to be kept at room temperature freeze drying carries the least risk of denaturation of the tissue proteins. It is not yet certain which technique is best suited to the particular requirements of the tissue bank. Methods which have been used include :—

(1) Simple freeze drying without control of the drying temperature of the material, as in small scale plasma drying. Normally a glass condenser is used.

(2) Freeze drying with a similar container but keeping the material at a known temperature, usually

Vol
3
195

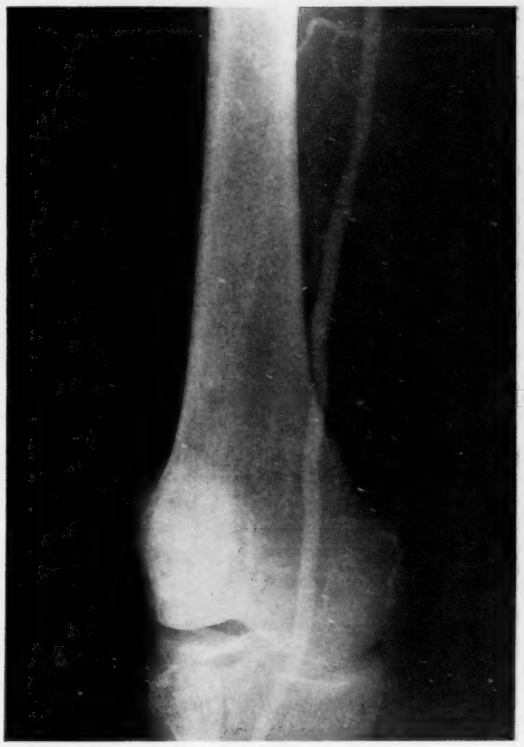


Fig. 1 (b). A stored homograft has been inserted; the blood supply has returned to normal, and with it the capacity for all normal activity (compare with Fig. 1a).

below its eutectic point, while drying.

(3) Freeze drying on a large scale using metal manifolds on oven-type stages.

Which method should be used for a particular tissue depends upon its physical structure in relation to the known phenomena of freeze drying. For example, some special tissues such as bone and fat contain a large proportion of non-cellular material which is much lower in water content than protein. To dry this material is often difficult and the process may need to be prolonged. Some means must then be sought to control the drying temperature which otherwise might rise quickly, since the total mass of material is great in relation to the available cooling effect of the tissue water during drying.

Histologists were among the first to apply freeze drying to the preparation and preservation of tissues. They have found that cellular detail is well retained and in some processes, for example fixation for histochemical and electron microscopic study, this is the method of choice[14]. In most instances the drying temperature is fixed at about –40°C. This greatly prolongs the drying time, a practical disadvantage, if the moisture is to be trapped by condensing it over solid carbon dioxide and alcohol at –79°C. This form of refrigeration is inconvenient for prolonged drying and a mechanical method, though it gives a temperature which is seldom lower than –30°C, is greatly to be preferred. The latter method if adopted can be usefully combined with a similar cooling circuit for the drying chamber. An arrangement of this kind may be the one best suited to the freeze drying of bone in all but small quantity. But specially designed apparatus is needed and is being developed in collaboration between the medical and technical specialists (Fig. 3).

Skin can be taken from cadavers in considerable quantities, though the surface area is great in proportion to the weight of the graft. This greatly favours freeze drying, which can therefore be completed easily and quickly, probably without the need for control of the drying temperature.

(b) The Method Employed at St. Mary's Hospital

As stated before arteries are ideally suited to preservation by freeze drying. They are a good example of how a valuable but perishable protein substance can be converted to a form, in which it is stable and easily transportable, yet at all times ready for use. At St. Mary's Hospital, London, we have been studying techniques for artery banking by freeze drying and have now used such grafts in patients on many occasions. The early results are comparable with those of using frozen grafts. So far, our method has been to freeze the arteries chosen for grafts in sterile Pyrex tubes with rubber caps. The tubes used are $\frac{3}{4}$ inch – $1\frac{1}{4}$ inch. in diameter with a wall thickness of approximately 1.5 mm. When the tubes were

immersed in a solid carbon dioxide-alcohol freezing mixture at –70°C or lower, pieces of human aorta and femoral artery varying in length between 5 and 30 cm. and in weight between 5 and 15 g. were found (by thermocouples) to reach –60° in about 15 minutes. Much more rapid chilling was obtained by immersing

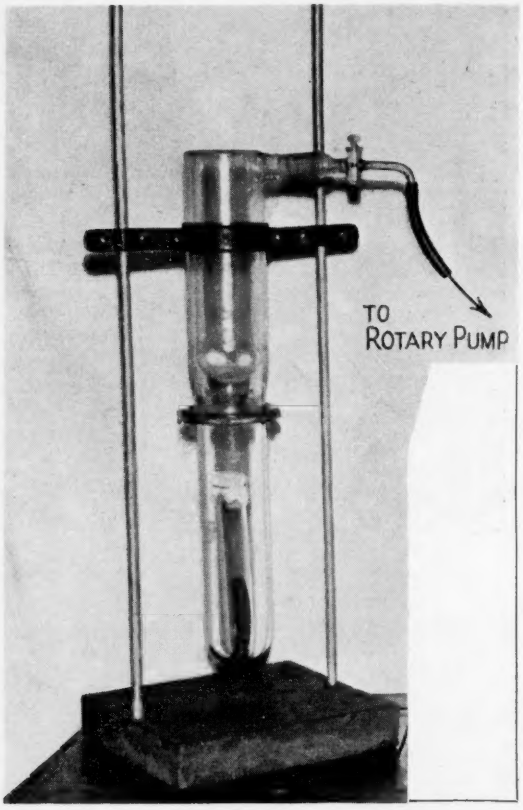


Fig. 2. Apparatus used at St. Mary's Hospital, London, for the primary freeze drying of arterial grafts. The wide-necked drying container permits 6-8 grafts in their own tubes to be dried at once; the short, wide path from the grafts to the condensing surface has been found to be considerably more efficient than using a standard B.34 adapted condenser and flask.

the grafts themselves in liquid Freon; this was true even with bath temperatures as high as –30°C, which can be readily maintained by mechanical refrigeration.

After allowing sufficient time for the grafts to reach their lowest possible temperature they are transferred to the frozen artery bank where they are kept in air at –70°C or lower[7]. The next step is to dry the grafts for 6 – 10 hours in a vacuum produced by a rotary oil pump and a solid CO_2–type glass condenser, without cooling the drying chamber. Pirani gauge heads are included in the apparatus at two points : the drying tube and the pumping line. Initial readings at both points are generally 0.1 mm. Hg, falling to 0.03 mm. Hg at the end of primary drying. Following this, the grafts are further dried for several days in an evacuated desiccator over phosphorus pentoxide. They are then stored in sealed evacuated tubes. The grafts, after reconstitution *in vacuo*, using a sterile intravenous set filled with isotonic saline, regain a normal gross and microscopic appearance (Fig. 4) and can be sutured to the patient's artery without difficulty. If reconstituted at atmospheric pressure microscopical structural disturbances, probably artifacts, have been seen.

Studies of the elasticity of grafts both before and after freeze drying have shown no change from the normal pattern for arteries, though freeze drying may slightly increase the modulus. Residual moisture determinations have been made at various stages in the process of primary and secondary drying using a commercially available moisture determination apparatus, in which the small amount of water remaining is removed *in vacuo*, trapped, and re-vaporised, then to be measured manometrically. 5% and 2% are usual values after primary and secondary drying respectively. These results compare closely with those obtained in identical samples weighed to within 0.1 mg. on a Stanton AD_2 balance. This weighing also yielded the information that a totally dried artery specimen of 75 mg. took up moisture at a rate of 0.4 mg. per minute. These studies also indicate that little harm can have resulted from allowing the grafts to warm as their moisture content falls, for specimens which were taken at –6 to –10°C were found already to have lost between 80 and 90% of their original water content. None of these samples after reconstitution *in vacuo* showed recognisable histological signs of tissue damage. This work has been reported in full elsewhere[15].

Batches of arterial or other grafts can be dried in

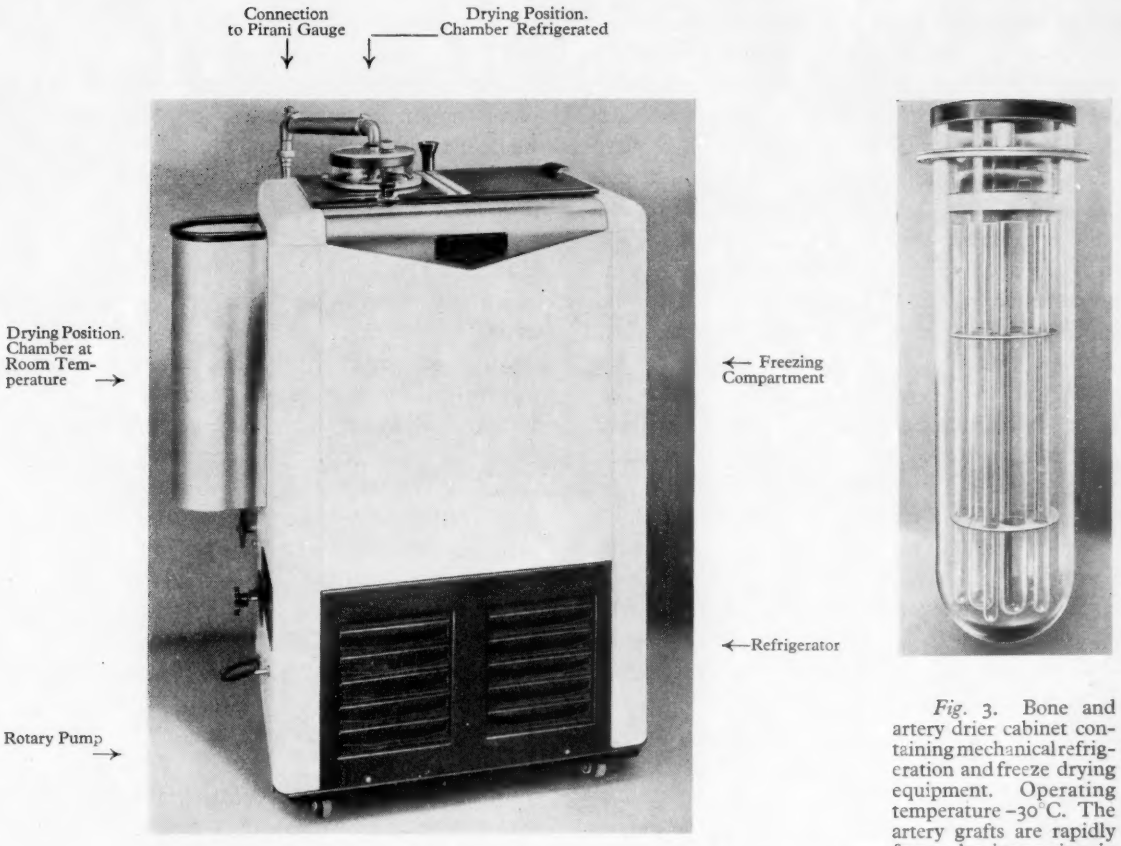


Fig. 3. Bone and artery drier cabinet containing mechanical refrigeration and freeze drying equipment. Operating temperature −30° C. The artery grafts are rapidly frozen by immersion in liquid Freon. The drying chamber is shown separately with the specimen tubes in position. The desiccant trays can be seen situated above the specimen tubes.

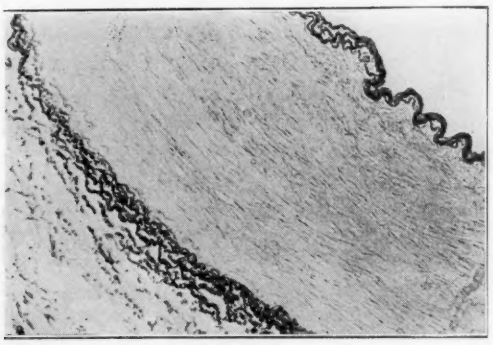
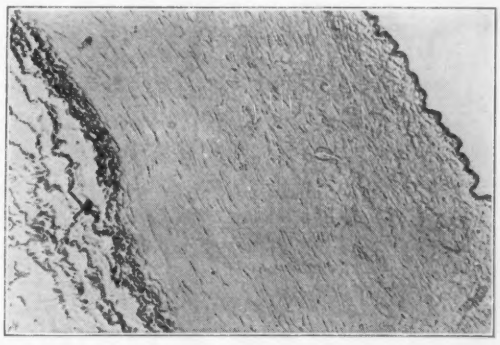


Fig. 4 (*a*). Section of fresh arterial graft, before freeze drying.



by courtesy of the Lancet

Fig. 4 (b). Section of same graft after freeze drying, and reconstitution in isotonic saline *in vacuo* (X 120, elastic tissue stain).

their original Pyrex tubes after replacing the rubber cap with a sterile gauze bacterial filter held in place by a rubber band, by placing them in a large, wide-bore drying tube (Fig. 2). The graft tubes are arranged to fit conveniently into the drying tube; their walls are in linear contact with one another and some with the inside of the drying tube. Using the wide-necked tube and condenser assembly the graft tube mouths are generally found to lie within 5 cm. of the condensing surface. This is fitted to the condenser unit by a large flat-flange vacuum joint. Up to 50 g. of artery have been dried in one day by this means. With such large batches it may be difficult to be sure that primary drying is complete. Apart from extrapolation from previous experience with similar batches the criteria are as follows: (*a*) The moisture content must be low when the rate, at which the carbon dioxide in the coolant solution is consumed, decreases, (*b*) when the type of fluorescence produced by a high frequency tester is similar in both the ' wet ' and ' dry ' parts of the system, and (*c*) when a Pirani gauge placed in the outflow from the condenser shows little tendency to register a rise in pressure, when the condenser is opened to the pump line after a period of isolation. At this stage the graft tubes, now no longer cold to the touch, are quickly transferred to a large vacuum desiccator, where they are supported horizontally above a tray of fresh phosphorus pentoxide, and the desiccator is evacuated to 0.05 mm. Hg or lower as measured by a Pirani gauge.

At least 3 days are allowed for this secondary drying; longer periods are more usual. In practice, as with the stage of preliminary freezing, its duration may largely depend upon when the opportunity arises to continue with the process. The graft tubes are finally sealed by replacing the gauze filters by specially designed Suba-seal caps through which the air is removed by a hypodermic needle connected to the pump. The seal is then completed with Apezion vacuum wax. No failure has been experienced with this type of closure during periods up to 9 months. A glass-sealed ampoule is clearly to be preferred, particularly in the case of grafts for military use. The walls of such containers must necessarily be thick; glass sealing under vacuum then presents some practical difficulty.

Outlook

We may expect to see a wider application of freeze drying to the needs of the tissue bank. Further developments are awaited in some important technical aspects of the process; notably in the achievement of sterility. Many organisms are killed by unprotected freeze drying, particularly after the long storage periods which are normal with this method of banking. Hufnagel[16] has found that by treating the grafts with ethylene oxide (a volatile disinfectant used in the preparation of bacteriological media) it is possible to render them sterile even though they have been taken from the donor without aseptic precautions. He has also had experience of using animal arterial grafts in man after such treatment and believes that in some ways they are better tolerated by the host than fresh human grafts.

So far mammalian tissues, though they can now in many instances be frozen without harm, have not been shown to survive freeze drying. This may be the reason why corneal grafts preserved in this way give poor results. Skin resists drying to a greater degree than many tissues[17] but true freeze drying to a very low final moisture content has not yet been shown to be compatible with survival of any normal animal tissue. It may, however, be possible in future to overcome this difficulty; if so, the already large field of application of tissue drying may come to be extended into that now held by refrigeration and deep freezing.

References

1 LOEB, L., *The Biological Basis of Individuality*. (Charles C. Thomas, Springfield, Ill, 1945.)

2 MEDAWAR, P. B., *J. Anat.*, (*London*), **78,** (1944), 176-199.
'Behaviour and Fate of Skin Autografts and Skin Homografts in Rabbits'.

3 DEMPSTER, W. J., *Brit. Med. J.*, **2,** (1951), 1041-1049.
'Problems Involved in Homotransplantation of Tissues with Particular Reference to Skin'.

4 SAWYER, P. N. and PATE, J. W., *Surgery*, **34,** (1953)' 491-500.
'Bioelectric Phenomena as Etiological Agents in Intravascular Thrombosis'.

5 WILSON, P. D., *J. Bone Jt. Surg.*, **33A,** (1951), 307-323.
'Follow-Up Study of Use of Refrigerated Homogeneous Bone Transplants in Orthopaedic Operations'.

6 EASTCOTT, H. H. G. and HUFNAGEL, C. A., *Surg. Forum, Amer. Coll. Surg.*, (1950), 269-274.
'Preservation of Arterial Grafts by Freezing'.

7 EASTCOTT, H. H. G., *Ann. Roy. Coll. Surg.*, **13,** (1953), 177-198.
'Arterial Grafting for Ischaemic Lower Limb'.

8 POLGE, C., SMITH, A. U. and PARKES, A. S., *Nature*, **164,** (1949), 666.
'Revival of Spermatozoa after Vitrification and Dehydration at Low Temperature'.

9 MOLLISON, P. L. and SLOVITER, H. A., *Lancet*, **2,** (1951), 862-864.
'Successful Transfusion of Previously Frozen Red Cells'.

10 EASTCOTT, H. H. G., CROSS, A. G., LEIGH, A. G. and NORTH, D. P., *Lancet*, **1,** (1954), 237-239.
'Preservation of Corneal Grafts by Freezing'.

11 MEEKER, I. A. and GROSS, R. E., *Science*, **114,** (1951), 283-285.
'Low-Temperature Sterilization of Organic Tissue by High-Voltage Cathode-Ray Irradiation'.

12 BRASCH, A. and HUBER, W., *Science*, **105,** (1947), 'Ultrashort Application Time of Penetrating Electrons: A Tool for Sterilization and Preservation of Food in the Raw State'.

13 STRONG, W. R., *CIBA Foundation Symposium*, 1953, (J. & A. Churchill Ltd., London, 1954), 220-229.
'The Tissue Bank, its Operation and Management'.

14 GERSH, I. and STEPHENSON, J. L., *Biological Applications of Freezing and Drying*, (Academic Press Inc., New York, 1954).
'Freezing and Drying of Tissues for Morphological and Histochemical Studies'.

15 EASTCOTT, H. H. G., HOLT, L. B., PEACOCK, J. H. and ROB, C. G., *Lancet*, **1,** (1954), 1311-1314.
'Preservation of Arterial Grafts by Freeze Drying : A Simplified Method'.

16 HUFNAGEL, C. A., *CIBA Foundation Symposium*, 1953 (J. & A. Churchill Ltd., London, 1954), 220-229.

17 MEDAWAR, P. B. and BILLINGHAM, R. E., *Inst. Biol.* (*London*), (1951), 55-62. Symposium on Freezing and Drying.
'The Viability of Mammalian Skin after Freezing, Thawing and Freeze Drying'.

Vol. 3 1953

LETTERS TO THE EDITOR

An Automatic Toepler Pump and a Circulating Pump

Sommaire

DESCRIPTION D'UNE POMPE Toepler automatique comprenant un disque en verre fritté scellé dans le verre et recouvert d'une couche de mercure en remplacement de la vanne anti-retour conventionelle. Le disque peut résister à une différence de pressions supérieure à une atmosphère. Le vide limité obtenable avec la pompe est de l'ordre de 0.1 mm. Hg. Le deuxième type de pompe est également descrit, lequel utilise aussi un disque en verre fritté comme valve. La vitesse de pompage de cette dernière est ½ l./h. Cette pompe est utile quand la vitesse de purification d'un gaz est faible.

Toepler Pump

TO ATTAIN THE complete transference of a gas from one part of a vacuum system to another depends upon the physical properties of the gas in question. With gases like carbon dioxide and xenon, the conventional liquid air traps can perform the function rapidly. Less condensible gases such as nitrogen, krypton, argon can be transferred rapidly and efficiently by adsorption on activated charcoal cooled with liquid air. The least condensible gases, helium, hydrogen, neon, on the other hand, require some form of Toepler pump.

The illustrated automatic Toepler pump is unique in that a sintered glass disc sealed into glass and covered with a pool of mercury replaces the conventional non-return valve, *viz.*, a metal or glass ball-bearing holding a pool of mercury against a ground glass seating. A common failure with the ball bearing valve is the inability of the ball bearing to retain the pool of mercury, when the pressure difference above and below the valve exceeds about half an atmosphere. The sintered glass disc, on the other hand, is capable of withstanding a pressure difference greater than one atmosphere.

Convenient dimensions for this automatic Toepler pump are a 250 c.c. bulb H, a 4 mm. i.d. by-pass tube B, a No. 4 porosity-sintered disc sealed into 6 mm. i.d. glass tubing and covered with a pool of mercury 1.5 mm. depth. The electrical contacts are No. 18 s.w.g. tungsten, the top one enclosed in a sheath of glass with only the tip exposed just below the disc. The mercury reservoir has access to air or a vacuum

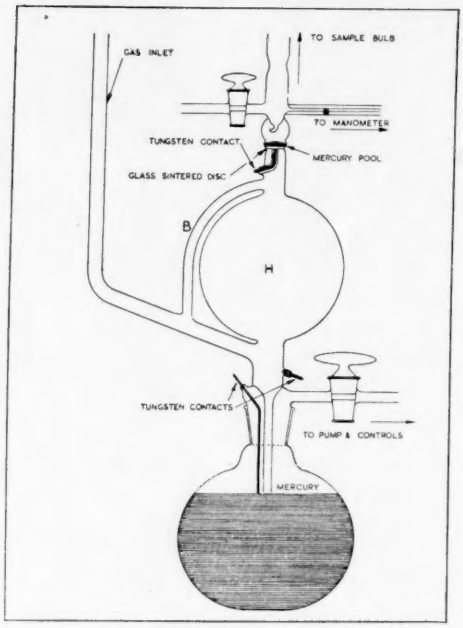


Fig. 1.

pump through one or two vacuum-tight solenoids containing spring-loaded plungers, and energising of the solenoids is operated through a relay connected to the tungsten terminals of the pump.

An automatic Toepler pump with the dimensions described above has been in operation for the past two years in the sampling of tritium, krypton 85 and helium, and no fracture of the sintered disc has occurred during that time[1]. The ultimate pressure to which the system can be evacuated, of course, depends on the dimensions of the pump, but with the one illustrated pressures down to 0.1 mm. Hg have been accomplished. In addition to pumping gas at pressures up to 20 cm. Hg or more, the pump can equally circulate a gas through a circuit of purification agents.

Circulating Pump

The pump illustrated in Fig. 2 is a 500 c.c. Pyrex glass bulb incorporating two sintered glass discs as described above. A large coil of Nichrome tape is sealed into the bulb through two tungsten-glass seals, and electric current is supplied through a step-down transformer and Simmerstat providing 35 watt in cycles of about one minute. The filament is heated to a temperature of about 500°C which causes some

Vol
3
195

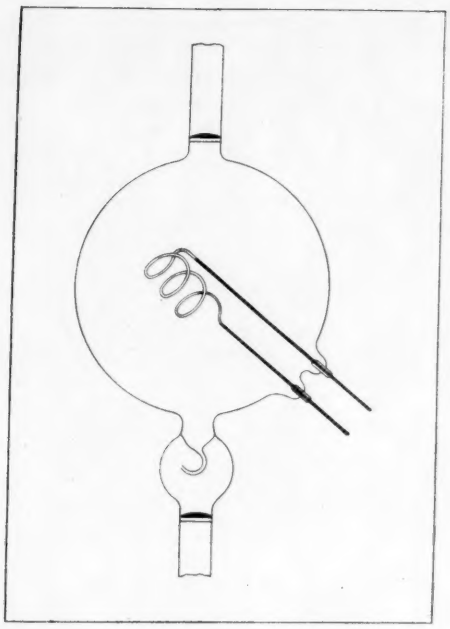


Fig. 2.

of the gas in the bulb to be expelled through the top non-return valve. When the filament cools, the gas is drawn through the lower valve. Pumping speeds of the order of 0.5 l./hr. are attained.

Though the speed of this pump is low, this is advantageous when the rate of purification of a mixture of gases is slow as in the separation of the fission rare gases during the dissolution of an irradiated uranium slug.

An apparatus, illustrated in the publication quoted describes how krypton 85 was separated from oxygen and certain hydrocarbons. The whole apparatus was maintained at atmospheric pressure using a cushion of helium, and the circulation pumps insured complete collection of the rare gases in activated charcoal cooled with liquid air.

E. J. WILSON.

Isotope Division,
Atomic Energy Research Establishment,
Harwell, Didcot,
Berks, England.

15th November, 1954.

Radioisotope Conference 1954 (Butterworths Scientific Publications, London, 1954), Vol. 2, 59-67:
W. J. Arrol, E. J. Wilson, C. Evans, J. Chadwick and J. Eakins.
'Preparation and Possible Industrial Uses of Krypton 85 and Tritium'.

A New Vacuum Society

Sommaire

ON POSAIT LA QUESTION dans un numéro recént de ce journal, si la formation d'une groupe de travail s'occupant avec la technique du vide creé au sein des institutions professionelles et scientifiques se trouverait utile. L'auteur indique que le développement de la science du vide a atteint le point où la formation d'une telle groupe serait en effet utile.

IN HIS ARTICLE in this journal, Mr. A. S. D. Barrett[1] gave a succinct and interesting account of those efforts which have been made abroad, for the advancement of the study and development of vacuum techniques and their applications, through the media of specialist journals, conventions and societies. He asked if there was a need in Great Britain for a society or some such body which will interest itself in vacuum work, and so provide a common meeting ground for the vast numbers of vacuum users.

It is impossible to give more than an opinion in answer to this question and difficult to assess the amount of active support such a society or group would receive. The ramifications of vacuum techniques are indeed widespread, and there is no foreseeable limitation to their further use and development, but to many users vacuum equipment provides little more than a service to processes which are often very diverse in characters. Such people will have few problems of common interest which cannot be aired if necessary in scientific or trade journals, or at their symposia organised by the learned societies, if only the latter were held more frequently.

The success of this journal may well be largely due to its novel and excellent form, but its increasing circulation, and within my own experience the continued demands for special courses in vacuum techniques, portends well for the future of this now mature branch of science. I believe despite those slight misgivings I have voiced, that as a start, a vacuum group within the framework of the various interested professional institutions would serve a useful purpose. Such a body would have the best opportunity to organise or sponsor meetings and attract support from interested persons not normally connected with vacuum.

D. W. STOPS.

Department of Applied Physics,
Northampton Polytechnic,
London, E.C.1, England.

1st February, 1955.

[1] BARRETT, A. S. D., *Vacuum*, 3, (1953), 1.

BOOK REVIEWS

Biological Applications of Freezing and Drying. Edited by R. J. C. Harris. (Academic Press Inc., New York, 1954). Pp. 415. Price £4 0s. 0d.

To-day there can be few research workers dealing with living cells and tissues or indeed with biological materials of any kind, to whom freezing and freeze drying are not of interest and importance. This new book is a most welcome and timely addition to the literature on the various and widening aspects of the subject. Within the covers of one volume of 14 chapters occupying some 400 pages is collected a series of up-to-date reviews written by acknowledged experts and pioneers in each particular field. The comprehensive bibliographies which conclude each chapter provide an outline of the history of the subject and an indispensable work of reference for the investigator concerned with the more recent developments.

It is appropriate that the first chapter, an excellent survey of 62 pages, should be devoted to the effects of low temperature, not only because of the importance of this subject in itself and in its various practical applications in the storage of cells, spermatozoa, etc. but also because freezing is an inseparable preliminary to freeze drying with which the major part of the book is concerned. Although this chapter deals mainly with the effect of low temperatures on living cells and tissues the physical principles involved are discussed in some detail. Following the notable advances made recently in the low temperature storage of various mammalian cells, special attention is given to the effect of additives, such as glycerol, on the salt/water relationship during freezing. The omission of the minus sign from the temperature scale in Fig. 12 reflects the error in the original paper from which the diagram is taken.

The development and the theory of freeze drying is dealt with in the next two chapters. The reader may be misled by the statement on page 70 into believing that all the human blood serum and plasma dried in this country during the second World War had been produced by the vacuum spin-freeze process, whereas it was in fact obtained by the use of the preliminary 'spin-freeze' method described in the previous paragraph. Exception must be taken to the conclusions drawn on page 72 on the drying of bacterial cultures by the method of Lord Stamp. The vacuum used is quoted as 100-300 micron instead of 100-300 mm. of mercury as stated in the original paper. Consequently the discussion as to whether or not freezing could take place during the drying loses its point. A true assessment of the situation is found later in the book, page 227, in the chapter on 'The Preservation of Bacteria'. In the section on vacuum measurement (page 111) it is acknowledged that the McLeod gauge is the accepted standard for measurements in the freeze drying range. A knowledge of its behaviour with an air–water system is therefore fundamental in any quantitative studies on the freeze drying process, and in the opinion of the reviewer a more detailed account of the errors inherent in this gauge under such conditions would be most desirable. It should be stressed that even though the dimensions of the gauge conform to the ideal requirements referred to, errors resulting from sorption of water on the glass of the gauge can be of considerable magnitude.

The next five chapters are devoted to special applications of freeze drying on the commercial or semi-commercial scale and deal with blood plasma and blood products, antibiotics, human milk, foodstuffs and finally culture media, 70 pages in all.

The preservation of viruses, of bacteria and of tissues each command a separate chapter. Here the ground is controversial and results are often conflicting especially in the case of mammalian cells and tissues where reliable experimental techniques are evidently most difficult to achieve. Those media found best for preserving bacteria do not necessarily prove so for viruses and one is struck by the apparent lack of any guiding principles on reading these two chapters. The reason no doubt is due to our almost complete ignorance of the mechanisms involved in maintaining viability in the dried state. A popular, though by no means unanimous, viewpoint is that optimal survival rates depend on the holding of an optimal (though small) amount of water in the dried sample, and even in the right places within the sample, and it may be hoped that at least one useful function this book may serve is to stimulate further enquiry into such matters.

With this important role of residual moisture in mind it is satisfying to find that a whole chapter has been devoted to the effects and measurement of residual moisture, a subject of unquestionable importance in freeze-dried materials generally quite apart from the special problem of the survival of viable cells. An especially elegant method developed recently for the measurement of minute amounts of

Vol
3
195

residual water is described in detail.

Chapters on the special application of freeze drying to electron microscopy and to the morphological and histochemical examination of tissues conclude the book.

The book as a whole is well produced and the reviewer has found only a small number of errors. The photographic reproductions however leave room for improvement.

B. R. RECORD.

. . . *of vacuum interest*

Lectures

United Kingdom. The Technical Optics Section of the Imperial College of Science and Technology organised a course of 8 lectures given by Dr. W. Weinstein on THIN FILM OPTICS which began on Tuesday, February 22nd 1955 and was held in the Physics Department of that College. The syllabus was:—

Part 1. General Theory

Maxwell's equations applied to multilayers.
Solution of the equation for reflection and transmission factors.
Periodic multilayers.
Continuously varying layers.
Graphical methods of computation.
Approximate methods.

Part 2 Applications

Anti-reflection coatings.
High-reflection coatings.
Beam splitters, neutral and dichroic.
Metal-dielectric interference filters.
All-dielectric interference filters.
Frustrated total reflection filters.

Part 3. Production and Control

Evaporation plant.
Measurement of film thickness.

Practical Course

Evaporation techniques.
Production of single and multilayer coatings, with examples to illustrate the lecture course.
Numerical calculations on the design of filters.
Testing of films.

The fee was 2 guineas for the lectures and 4 guineas for the practical course.

FURTHER LECTURES are announced as follows:—

December 16th, 1954 at 7 p.m.

Melting and Casting Under Reduced Pressure.
Lecturer : *H. H. Scholefield.*
Held at : Institute of Metals, London, S.W.1.
Arranged by : The Institute of British Foundrymen, London Section.

February 8th, 1955 at 5.30 p.m.

The Mechanism of Drying of Solids.
Lecturers : *E. W. Bruce, R. F. Strickland-Constable & D. M. Newitt.*
Held at : Geological Society, Burlington House, London, W.1.
Arranged by : Institution of Chemical Engineers, London Section.

February 21st, 1955 at 5.30 p.m.

Vacuum Fumigation: (*a*) Physico-Chemical Effects, (*b*) Biological Effects.
Lecturers : (*a*) *W. Burns-Brown,* (*b*) *A. P. B. Page.*
Held at : Chemical Society, Burlington House, London, W.1.
Arranged by : Society of Chemical Industry, Pesticides Group.

March 23rd, 1955 at 7 p.m.

High Vacuum Technique.
Lecturer : *T. W. Rowe.*
Held at : 5 King's Place, King Street, Chester.
Arranged by : Society of Instrument Technology, Chester Section.

April 20th, 1955 at 7 p.m.

Vacuum Evaporation Plant for Metallising Volatile Plastic Materials.
Lecturer : *L. Holland.*
Held at : St. Mark's House, 186 Woodhouse Lane, Leeds.
Arranged by : Plastics Institute, Yorkshire Section.

April 30th, 1955 at 10 a.m.

The Optical Properties of Some Multilayer Systems : (*a*) Theoretical, (*b*) experimental.
Lecturer : O. S. Heavens.
Held at : Zoological Theatre, University of Reading.
Arranged by : Royal Photographic Society, Scientific & Technical Group.

May 17th, 1955 at 3 p.m.

New Developments in the Field of High Vacuum Technique.
Lecturer : *O. Winkler.*
Held at : Great Hall of the Congress Building.
Arranged by : The Management of the ACHEMA Exhibition, Germany.

Holland. An International Symposium on Electrical Discharges in Gases was held at the Technical University of Delft, Holland, from the 25th-30th, April, 1955. Lectures were given on the following subjects: Fundamental Processes and New Views on the Mechanism of Gas Discharges. Instabilities and Conditions of Stability, Oscillations and Noise Phenomena in Gas Discharges. Breakdown Potential as a Function of Potential Difference and Frequency. New Methods of Measurement Applied to the Investigation of Gas Discharges. Gas Discharges Applied to Other Physical Problems as a Method of Measurement. Arc Discharges. Spark Discharges. The Secretary of the organising committee: A. W. van Wagensveld, Mijnbouwplein 11, Delft, Holland.

•

United States. The Committee on Vacuum Techniques Inc., Boston, Mass., U.S.A., plans to arrange for a second Symposium on Vacuum Technology to be held at Mellon Institute, Pittsburgh 13, Pa., which is to last three days beginning with Thursday, October 13th 1955. Those interested in contributing a paper should contact Mr. Rudy Koehler, President, Committee on Vacuum Techniques Inc., Box 1282, Boston 9, Mass., U.S.A.

Professional

April, 1955.

Belgium. A special working group to deal with vacuum engineering has been formed recently at the Inter-University Institute of Nuclear Science. The purpose of the group is to sponsor developments and improvements in vacuum technique through the various local centres of the Institute. The Chairman of the group is Dr. E. Thomas, Associate Professor at the Royal Military School and Joint Director of the Centre of Nuclear Physics at that school. The group wishes to make and maintain contact with similar organisations in other countries. All communications should be addressed to the Chairman 30, Avenue de la Renaissance, Bruxelles, Belgium.

Personal

December, 1954.

Dr. C. R. Burch, F.R.S., of Bristol University was awarded the Rumford Medal of the Royal Society for his outstanding contributions to the development of vacuum technology such as the creation of the oil diffusion pump which facilitated the design of high speed particle accelerators and other vacuum-operated apparatus requiring very high vacua.

•

April, 1955.

We hear that Mr. L. Holland well-known for his contributions to the art of vacuum coating has been awarded the Silver Medal of the Plastics Institute, London, for a paper which he has published recently on ' The Degassing Properties in Vacuum of Plastics Materials Metallised by Vacuum Evaporation '.

Books

The following books on vacuum subjects have been published recently :

1954

United States

Isotopic Gas Analysis for Biochemists.
Author : *R. F. Glascock.*
Published by : Academic Press Inc., New York.
pp. 247. Price £2 1s. 6d.
(to be reviewed in this journal)

United Kingdom

Zirconium.
Author : *G. L. Miller.*
Published by: Butterworths Scientific Publications, London.
pp. 381. Price £2 5s. 0d.
(to be reviewed in Vol. III No. 4)

Biological Applications of Freezing and Drying.
Edited by : *R. J. C. Harris.*
Published by : Academic Press Inc., New York.
pp. 415. Price £4 0s. 0d.
(reviewed in this issue)

1955

United Kingdom

Properties of Thin Films.
Author : *O. S. Heavens.*
Published by: Butterworths Scientific Publications, Ltd., London.
pp. 261. Price £1 10s. 0d.
(to be reviewed in Vol. IV No. 1)

VACUUM
CLASSIFIED ABSTRACTS

Vol.
3
1953

Vacuum Research, Apparatus and Instruments

The publishers endeavour to make the compilation of abstracts published in this section as comprehensive as possible, but cannot undertake to trace and quote all relevant information published on these subjects.

All abstracts are formulated strictly in accordance with the contents of the original publication, but the publishers are not responsible for such mistakes as may have been made in abstracting. Statements or opinions expressed in the publications quoted do not necessarily represent the experience and/or opinions of the publishers.

Arrangement of Subjects

All abstracts published in this section are allocated to one or more of the subjects shown in the following list:

(I) General Science and Engineering

10 — VACUUM ENGINEERING — GENERAL
11 —
12 —
13 — ELECTRICAL SCIENCES
14 — MECHANICAL SCIENCES
15 — PHYSICS
16 — CHEMISTRY
17 — METALLURGY
18 —
19 — SCIENCES (other than the above)

(II) Vacuum Apparatus and Auxiliaries

20 — PUMPS
21 — GAUGES
22 — MEASURING PLANT — VACUUM
23 —
24 —
25 —
26 — MATERIALS
27 —
28 — APPARATUS COMPONENTS
29 — MISCELLANEOUS APPARATUS

Vol. 3 1953

(III) Vacuum Processing Techniques

30 — EVAPORATION
31 — CATHODIC SPUTTERING
32 —
33 — IMPREGNATION
34 — FUMIGATION
35 —
36 — DRYING
37 — METALLURGICAL PROCESSES
38 — DISTILLATION
39 — MISCELLANEOUS PROCESSES

(IV) Special Subsidiary Subjects

40 — GASES & VAPOURS
41 — FREEZE DRYING
42 —
43 —
44 —
45 — ELECTRONICS
46 —
47 — GLASS PROCESSING
48 —

Indices

Three indices are appended to each issue of abstracts: (1) A Cumulative Subject Index in front of the text, (2) a Cumulative Author Index at the back of the text and (3) a List of Periodical Publications quoted. Index (1) and (2) will show all abstracted information published up-to-date in the current volume of the journal.

Filing of Abstracts

All pages containing the text of abstracts are printed on one side only and are detachable ready for filing in special folders. Each main group of subjects (I, II, III, IV) starts on a separate page in each issue and all abstracts in any one group are numbered consecutively throughout all issues of a volume. To distinguish the current numbers of abstracts belonging to one of the four groups from those allocated to other groups, the group number is shown as a suffix to the current number of the abstract, *e.g.*, 20/I as distinct from 20/II, etc.

Translations

Translations of some of the foreign publications quoted can be supplied.

Cumulative Subject Index, Vol. III (1953)

For information published in the first and second volumes under these or other headings see October 1951 and October 1952 issues respectively.

Subject — Abstract No.

I — General Science and Engineering — I

(For Abstract Nos. 1/I-68/I see January issue) (For Abstract Nos. 68/I-131/I see April issue)

10 — VACUUM ENGINEERING (GENERAL) — 10

Clean up Phenomena 134/I;
Conferences 69/I;
Contamination of Vacuum Systems ... 77/I; 138/I;
Degassing of Vacuum Systems and Vessels 78/I; 80/I; 81/I;
Developments 70/I;
Dry Boxes 73/I; 74/I;
Flow (Theory of—) 133/I;
Freeze Drying 4/I; 83/I; 84/I; 140/I; 141/I; 142/I;
Gas Evolution 79/I;
Gas Supply to Vacuum Systems 9/I; 82/I; 136/I; 137/I;
Gauges 7/I; 8/I;
Gettering 75/I; 76/I; 135/I;
Leak Detection 139/I;
Metallisation 1/I; 2/I; 85/I; 86/I; 143/I; 144/I; 145/I;
Miscellaneous Applications 10/I; 146/I; 147/I;
Professional Organisations 132/I;
Pumps 6/I; 72/I;
Residual Gases 138/I;
Surveys (General—) 3/I;
Vacuum Drying 83/I;
Vacuum Systems (Design of—) 71/I;
Vacuum Systems (General Testing of—) ... 5/I;

13 — ELECTRICAL SCIENCES — 13

Circuitry 16/I; 92/I; 149/I;
Condensers 17/I;
Discharges 11/I; 12/I; 13/I; 89/I; 90/I; 150/I; 151/I; 152/I;
Electrical High Vacuum Analogies 148/I;
Insulation in Vacuum 87/I; 88/I;
Lamps 93/I; 154/I; 155/I;
Power Generation 14/I;
Rectification 15/I;
Static Electrification 91/I;
Television 153/I; 156/I;

14 — MECHANICAL SCIENCES — 14

Bursting Discs (Steam Pressure) 96/I;
Sealing by Cold Welding 18/I;
Welding and Brazing (Vacuum-Tight—) ... 94/I; 95/I; 157/I;

15 — PHYSICS — 15

Astro-Physics 98/I;
Atomic 22/I;
Crystals (Metallic—) 31/I; 159/I; 160/I; 161/I;
Evaporation—General 33/I; 34/I; 35/I; 160/I;
Filters (Optical—) 23/I; 24/I; 25/I; 103/I; 164/I;
Infra-Red Radiation and Absorption ... 20/I; 103/I;
Low Temperature 107/I;
Luminescence 106/I;
Microscopy (Light—) 36/I; 105/I;
Optical—General 26/I; 27/I; 29/I; 100/I; 101/I; 103/I; 104/I; 162/I; 164/I; 165/I; 166/I;
Optical Methods of Measurement 104/I;

Vol 3 195

Cumulative Subject Index, Vol. III (1953)

Contd.

For information published in the first and second volumes under these or other headings see October 1951 and October 1952 issues respectively.

Subject	Abstract No.
Physics continued	
Permeability of Materials	38/I;
Photo-Conductivity	19/I; 163/I;
Radioactivity	21/I; 30/I; 97/I;
Spectroscopy (Infra-Red—)	102/I;
Sublimation	32/I; 33/I; 34/I; 35/I; 158/I;
Surface Studies of Solids	36/I;
Thermodynamics	37/I;
Ultra-Violet Photography	28/I;
Ultra-Violet (Vacuum—)	99/I; 100/I; 101/I; 167/I; 168/I;
Upper Atmosphere (Research of the—)	39/I; 97/I;
16 — CHEMISTRY — 16	
Alcohol	118/I;
Antibiotics	40/I;
Blood and Blood Plasma	41/I; 42/I; 173/I;
Catalytical	49/I; 110/I; 177/I; 178/I;
Caustic Soda	169/I;
Fats (Animal—)	117/I;
Fatty Acids	44/I; 46/I;
Fertilisers (Production of—)	170/I;
Food Preservation	108/I;
Historadiography	174/I;
Hydrocarbons	45/I; 119/I;
Magnesium Chloride	169/I;
Micro-Organisms	109/I; 113/I;
Moisture Determination	42/I; 43/I; 171/I;
Oils (Vegetable—)	44/I; 116/I; 117/I;
Organic Compounds	114/I;
Oxygen Determination in Organic Substances	176/I;
Polystyrene (The Degradation of—)	115/I;
Radio Chemistry	111/I;
Russell Effect	48/I; 179/I;
Silicon Monoxide	50/I; 51/I;
Spectro-Chemical Analysis of Gases	47/I;
Surgical and Clinical	41/I; 175/I;
Toxicological	172/I;
Tritiation of Organic Compounds	112/I;
17 — METALLURGY — 17	
Beryllium	124/I;
Chromium	61/I; 126/I;
Coating of Metals (Various Methods of—)	123/I;
Corrosion of Metals	61/I;
Developments	180/I; 182/I;
Gas Content of Melts (Determination of the—)	58/I; 184/I;
Iron and Steel	125/I;
Lost-Wax Process	63/I;
Metal Oxides (Chemical Changes in Vacuum Processed—)	62/I;
Molybdenum	53/I; 55/I; 124/I; 129/I; 183/I;
Oxidation of Metals	54/I;
Oxygen Solubility in Metals	125/I;
Powder Metallurgy	64/I;
Protective Atmosphere Furnaces	55/I; 183/I;
Sealing Porous Castings	65/I; 122/I;
Spectra of Vaporised Metals	185/I;
Structure of Metals	59/I; 60/I; 64/I; 121/I;
Tin	66/I;
Titanium	56/I; 57/I; 124/I; 127/I;
Tungsten	53/I; 130/I;
Vapour Pressure Chart	52/I;
Zinc	120/I;
Zirconium	124/I; 128/I; 181/I;

Cumulative Subject Index, Vol. III (1953)

Contd.

For information published in the first and second volumes under these or other headings see October 1951 and October 1952 issues respectively.

Subject	Abstract No.

19 — SCIENCES (other than the above) — 19

Building Industry (Vacuum Processing in the—)	67/I;
Dental	186/I;
Films by Non-Vacuum Methods (Deposition of—)	131/I;
Insect Flight (Study of the Dynamics of—)	68/I;

II — Vacuum Apparatus and Auxiliaries — II

(For Abstract Nos. 1/II-40/II see January issue) (For Abstract Nos. 41/II-84/II see April issue)

20 — PUMPS — 20

Booster Pump	88/II; 89/II;
Circulating Pump	3/II;
Diffusion Pump	41/II; 42/II; 43/II; 86/II; 87/II; 89/II;
Fluids (Pump—)	6/II; 7/II; 90/II;
Gas-Ballast Pump	4/II;
Ionisation Pump	1/II; 2/II; 85/II;
Jet Pump	44/II; 45/II;
Measurement of Pump Performance ...	91/II;
Oil Ejector Pump	4/II;
Purification of Pump Fluids	6/II; 7/II;
Rotary Pump	4/II; 5/II; 6/II; 7/II; 46/II;
Various Pumping Techniques	8/II;
Water Ring Pump	92/II;

21 — GAUGES — 21

Aneroid Capsule Gauge	98/II;
Developments	10/II;
Diaphragm Manometer	48/II; 49/II;
Ionisation Gauge	14/II; 52/II; 96/II;
McLeod Gauge	11/II; 93/II;
Mercury Column Manometer	47/II; 93/II;
Phillips-Type Gauge	50/II; 51/II; 97/II;
Pirani-Type Gauge	12/II; 13/II; 95/II; 96/II;
Pressure Measurement (Various Methods of—)	9/II;
Quartz-Fibre Manometer	94/II;

22 — MEASURING PLANT (VACUUM) — 22

Electron Diffraction	15/II; 16/II; 18/II; 56/II; 58/II; 62/II; 103/II;
Electron Microscopy	18/II; 19/II; 55/II; 56/II; 57/II; 58/II; 104/II; 105/II; 106/II;
Field Emission Microscope	53/II;
Mass Spectrometer	20/II; 21/II; 22/II; 59/II; 60/II; 61/II; 100/II; 101/II; 102/II;
Specimen Preparation	17/II; 54/II; 55/II; 103/II; 104/II; 105/II; 106/II; 107/II;
X-Ray Apparatus	99/II;

Vol 3 195

Cumulative Subject Index, Vol. III (1953)
Contd.

For information published in the first and second volumes under these or other headings see October 1951 and October 1952 issues respectively.

Subject	Abstract No.
26 — MATERIALS — 26	
Ammonia	63/II; 110/II;
Carbon and Carbon Products	24/II; 25/II; 26/II; 27/II;
Ceramics	24/II;
Dry Ice	111/II;
Drying Agents	64/II;
Glass	23/II; 113/II;
Inconel	31/II; 32/II;
Mercury	30/II; 114/II; 115/II;
Paper	112/II;
Plastics	66/II;
Porcelain	108/II;
Properties of Materials (Vacuum—)	24/II; 65/II;
Refractories	29/II; 67/II; 109/II;
Refrigerants	63/II; 110/II; 111/II;
Rubber	116/II;
Silicon Rubber	28/II;
Stainless Steel	68/II;
Teflon	28/II;
28 — APPARATUS COMPONENTS — 28	
Filters (Mechanical and Chemical—)	70/II;
Gaskets	71/II;
Joints	33/II; 73/II; 74/II; 75/II;
Leaks (Controlled—)	69/II;
Locks	77/II; 78/II; 79/II;
Seals (Other than Shaft—)	34/II; 72/II;
Traps	80/II;
Valves (Mechanical—)	35/II; 36/II; 76/II; 117/II; 118/II; 119/II; 120/II; 121/II;
29 — MISCELLANEOUS APPARATUS — 29	
Crystalliser	128/II;
Flow Control (Gas—)	38/II;
Gases (Storage of Liquefied—)	126/II;
High Altitude Rockets	37/II;
High Altitude Test Chambers	83/II;
Leak Detector	123/II; 124/II;
Liquefier (Gas—)	39/II; 125/II;
Pressure Control	82/II; 122/II;
Safety Devices	81/II; 127/II;
Vapour Pressure of Materials (Determination of—)	40/II; 84/II;

III — Vacuum Processing Techniques — III

(For Abstract Nos. 1/III-52/III see January issue) (For Abstract Nos. 53/III-96/III see April issue)

30 — EVAPORATION — 30	
Aluminising of T.V. Tubes	62/III; 105/III;
Aluminium	6/III; 55/III; 59/III;
Conducting Films (Electricity—)	13/III; 63/III;
Contamination of Films	7/III; 58/III;
Continuous Band (Coating of—)	16/III; 17/III;
Crucible Techniques	9/III; 56/III; 59/III; 60/III; 99/III; 100/III;
Germanium	100/III;
Glass (Deposition of Metal on—)	11/III; 24/III;
Graticules (Manufacture of—)	110/III;

Cumulative Subject Index, Vol. III (1953)

Contd.

For information published in the first and second volumes under these or other headings see October 1951 and October 1952 issues respectively

Subject	Abstract No.
Evaporation Continued	
Light Absorbing Films	12/III; 66/III; 109/III;
Metals (Deposition of Common—)	3/III; 4/III; 20/III;
Metals (Deposition of Precious—)	61/III; 102/III; 111/III;
Multiple-Layer Films	8/III; 9/III; 10/III; 13/III; 108/III;
Oxide Films	5/III; 13/III; 23/III; 66/III; 104/III;
Photo-Sensitive Layers	107/III;
Plant (Various Designs of—)	9/III; 56/III;
Plastics (Deposition of Metal on—)	2/III; 3/III; 4/III; 16/III; 53/III; 55/III; 114/III; 115/III;
Properties of Films (Electrical—)	20/III; 21/III; 22/III; 64/III;
Properties of Films (Optical—)	19/III; 67/III; 68/III; 109/III;
Reflecting Films	61/III;
Reflection-Reducing Films	8/III; 14/III;
Replicas Techniques (Various—)	26/III; 104/III;
Salts	6/III;
Shadow Casting	25/III; 26/III; 101/III;
Silicon and Silicon Compound Films	22/III; 27/III;
Sorption of Gases by Films	18/III; 112/III; 113/III;
Structure of Films	15/III; 97/III; 102/III; 103/III;
Substrate Materials	24/III;
Thickness of Films (Measurement of—)	19/III; 57/III; 109/III;
Thin Films—General	1/III; 53/III; 54/III; 97/III; 98/III;
Thin Films—Miscellaneous Applications	65/III;
Titanium (Thin Films of—)	106/III;

31 — CATHODIC SPUTTERING — 31

Conducting Films (Electricity—)	30/III; 69/III; 70/III;
Metals (Deposition of Precious—)	116/III;
Oxide Films	30/III; 70/III;
Photo-Sensitive Layers	29/III;
Physics of Sputtering	28/III;
Properties of Films (Electrical—)	70/III;
Thin Films—General	31/III;

33 — IMPREGNATION — 33

Castings	32/III; 71/III;
Cops (Carpet Making)	117/III;

34 — FUMIGATION — 34

Developments	72/III; 73/III;

36 — DRYING — 36

Concentration of Liquids (Methods)	77/III; 118/III; 119/III;
Drying—General	33/III; 74/III;
Evaporators	33/III; 77/III; 78/III; 119/III,
Meat	75/III;
Puff-Drying	76/III;
Theory and Design	34/III;

Cumulative Subject Index, Vol. III (1953)

Contd.

For information published in the first and second volumes under these or other headings see October 1951 and October 1952 issues respectively.

Subject	Abstract No.
37 — METALLURGICAL PROCESSES — 37	
Developments	120/III; 121/III;
Diffusion (Coating of Metals by—)	83/III;
Distillation of Metals	37/III;
Etching	36/III;
Furnaces and Ovens	39/III; 40/III; 41/III; 42/III; 79/III; 121/III; 122/III; 123/III; 124/III; 125/III; 126/III; 127/III;
Fusion Methods (Vacuum—)	85/III; 128/III;
Hot Extraction Methods	85/III;
Melting and Casting	39/III; 80/III; 84/III;
Oxygen Determination	128/III;
' Rare ' Metals (Processing of—)	38/III; 41/III; 81/III; 82/III;
Reduction of Metallic Oxides	84/III;
Separation of Metals	37/III;
Sintering	35/III;
38 — DISTILLATION — 38	
Centrifugal Still	87/III;
Column Still	43/III; 44/III; 87/III;
Pot Still	45/III;
Sea Water	130/III;
Stills (Various Types of—) and Auxiliary Equipment	86/III; 88/III; 130/III;
Vapour-Liquid Equilibria	129/III;
39 — MISCELLANEOUS PROCESSES — 39	
Carbon^{14} (A Method of Handling—)	91/III;
Concrete (The Setting of—)	48/III;
De-Icing of Glass Panels	46/III;
De-Odorisation	51/III; 52/III; 94/III;
De-Oiling of Fruit Juices	93/III;
Drilling by Electrons	132/III;
Food Packaging	96/III;
Food Processing	49/III; 50/III; 92/III; 93/III; 95/III; 134/III; 135/III;
Geological Age Determinations	90/III;
Magnesium Targets (Preparation of—)	47/III;
Potash Refining	133/III;
Refrigeration (Vacuum—)	49/III;
Thermal Insulation (Vacuum as a Means of—)	89/III; 131/III;
IV — Special Subsidiary Subjects — IV	
(For Abstract Nos. 1/IV-18/IV see January issue) (For Abstract Nos. 19/IV-37/IV see April issue)	
40 — GASES AND VAPOURS — 40	
Analysis	3/IV; 21/IV; 39/IV;
Blending (See Filling, Mixing and Blending)	
Carbon Dioxide	2/IV;
Conductivity (Thermal—)	4/IV;
Filling, Mixing and Blending	21/IV; 39/IV;
Flow	6/IV; 38/IV;
Hydrogen	19/IV; 44/IV; 45/IV;
Ionisation	24/IV; 25/IV;
Isotopic Gases	22/IV; 23/IV;
Liquefaction	7/IV; 42/IV;
Oxygen	46/IV;
Permeation	5/IV; 44/IV;
Purification	23/IV;
Rare Gases	20/IV; 23/IV; 43/IV;
Sorption of Gases	40/IV; 41/IV;

Vol. 3 1953

Cumulative Subject Index, Vol. III (1953)

Contd.

For information published in the first and second volumes under these or other headings see October 1951 and October 1952 issues respectively.

Subject	Abstract No.

41 — FREEZE DRYING — 41

Apparatus and Methods (Various—)	53/IV;
BCG Vaccine	29/IV;
Biological Materials (Various—)	48/IV;
Blood	10/IV; 27/IV; 49/IV;
Developments	48/IV;
Food	30/IV;
Freeze Drying—General	26/IV; 49/IV;
Gelatine	9/IV;
Heating Systems	12/IV;
Ice Sublimation	47/IV;
Quality Control	10/IV;
Rinderpest Virus	53/IV;
Streptomycin	13/IV;
Survival of Bacteria (Studies of the—)	29/IV;
Theory and Design	8/IV;
Tissue	11/IV; 28/IV; 50/IV; 51/IV;
Tobacco Mosaic Virus	52/IV;

45 — ELECTRONICS — 45

Charged Particle Accelerators	16/IV; 17/IV; 37/IV; 38/IV; 54/IV;
'Clean Up'—Phenomena	55/IV;
Field Emission	56/IV;
L-Cathode	36/IV;
Oxide Coated Cathode	34/IV; 35/IV; 36/IV;
Secondary Emission	33/IV; 34/IV; 35/IV;
Vacuum Breakdown Phenomena	15/IV; 31/IV; 32/IV;
Valves (Electronic—)	14/IV;
Work Function and Contact Potential	57/IV; 58/IV;

47 — GLASS PROCESSING — 47

Bonding to Other Materials	59/IV;
Glass-to-Metal Seals	18/IV; 60/IV;

Vol
3
19

I — General Science and Engineering — I

Abstract No. and References

10 — VACUUM ENGINEERING (GENERAL) — 10

The Thoughts and Actions Leading to the Formation of the C.V.T.

132/I
Letter by J. H. Durant
Vacuum
3, April 1953
162-163

United States. The chairman of the sub-committee on publicity and publication of the Committee on Vacuum Techniques, Inc. gives a detailed account of the events which led to the formation of this new organisation.

Sommaire : Bref exposé des événements ayant précédé la formation du 'Committee on Vacuum Techniques'.

An Electrical Analogue to a High Vacuum System

133/I

See Abstract No. : 148/I

The Electronic Clean-Up of Gases in Sealed-Off Vacuum Systems

134/I

United Kingdom. Some of the previous work in this field is reviewed briefly, and a few of the conflicting theories put forward to account for the observations are mentioned. The results of fresh experiments, using sealed-off tungsten filament triode ionisation gauges, are described. In the cleaning-up of gas in these tubes, two regions of the log P *vs.* time curves are distinguished. The speed of pumping or cleaning-up of gas in each region is constant, and hence the rate of cleaning-up of gas is proportional to pressure. In the first or ' A ' region of the falling pressure/time curves, the speed of pumping is a few cm.^{3} sec.$^{-1}$; in the second or ' B ' region, the speed is about 1/20th as great. In the ' A ' region, the speed of pumping is proportional to the ionising electron current, but in the ' B ' region it is independent of electron current for values greater than a few tens of microamperes. It has been found that the cleaned-up gas is held by the glass walls of the tubes, particularly in the vicinity of the electrode structure. When all the glass was shielded from the products of the ionising electron current, clean-up of gas ceased. The significance of the two regions of the clean-up is discussed, and possible mechanisms are suggested in keeping with the observations in the greater part of the present experiments, and those reported by previous workers. (*Authors*)

Article by R. N. Bloomer & M. E. Haine
Vacuum
3, April 1953
128-135

Sommaire : Le phenomène d'abbaissement de la pression a été étudié expérimentalement en utilisant des jauges à ionisation à triodes scellées, à filaments de tungstène.

Sorption of Gases at Very Low Pressures by Thorium Powder

135/I

United Kingdom. Thorium powder is frequently used as a getter in thermionic valves. The author investigated the physico-chemical processes on which the gettering action of thorium is based, using an experimental arrangement for measuring the rates of sorption which was described previously (see Vol. I, Abstract No. 23/I). The measurements were carried out in the pressure range of 10^{-7}–10^{-3} mm. Hg. The thorium powder employed had a particle size of 1–10 μ and was deposited electrophoretically on oxide-cathode sleeves made from nickel with an area of 2 cm.^{2} giving a coating of 5 mg. weight. After coating the sleeves were outgassed and slightly sintered in vacuum. The rate of sorption for oxygen was found to be high but only over a short period, following which the sorption rate decreased, rapidly at first and more gradually later. This decay became more pronounced at higher pressures and lower temperatures. The rate of sorption for hydrogen is smaller than that of oxygen. It increases with temperature up to a maximum at about 750°K and decreases at higher temperatures. Investigating the reversibility of the process it was found that, in the case of oxygen, heat treatment of the thorium at 1,150°K permitted full recovery of the amount originally sorbed, only where the quantities sorbed did not exceed approximately 1 $\mu \times$ l. In the case of hydrogen however the process could be reversed completely by a heat treatment of the thorium lasting 5 minutes. It was concluded from these experiments that oxygen is gettered by a mechanism of chemisorption resulting in the formation of a stable oxide, likely to be ThO_2, with a low dissociation pressure. Investigation of the temperature dependence of oxygen sorption gave further proof of the chemical nature of the process and yielded the value of 0.75 Kcal/mol for the activation energy of the thorium samples used in the experiments. But the sorption of hydrogen is a process of solution and facilitates the sorption and re-evolution of the gas as required. During the determinations of the oxygen sorption rates the presence of a small residual pressure above the thorium was noticed, prior to the admission of oxygen. This was suspected to be hydrogen as according to Sieverts and Roell the amounts of hydrogen dissolved in thorium are correlated thermodynamically with the hydrogen pressure outside the metal and thus hydrogen gas could be present, if it was not previously completely removed from the metal during the heat treatment. This was proved to be correct in experiments with a modified arrangement, described in detail and involving the use of a palladium tube. Tests in accordance with a method proposed by Langmuir indicated that the re-evolved hydrogen is atomic and will recombine into molecules only after some time. A theoretical study of these conditions follows. The total quantities sorbed as a function of temperature have been determined as shown in Tables 4 and 5 reproduced below :

Table IV. Total Quantities of Oxygen Sorbed in Thorium

Temperature during Sorption (°K)	350	950	950
Sorption Rate at Beginning (cm.^{3}/sec.)	560	650	970
Total Sorbed Quantity (μ x l.)	0.7	21	26*

* Refers to 10 mg. thorium.

Vol. 3 1953

I — General Science and Engineering — I
Contd.

Abstract No. and References

Table V. Total Quantities of Hydrogen Sorbed in Thorium

Temperature during Sorption (°K)	500	565	630	740	845	935
Total Sorbed Quantity (μ x l.)	1.08	1.42	2.12	1.98	0.73	0.34

The results of the investigation are summarised as follows : The rate of sorption of oxygen is high even at very low pressures. Oxygen sorption is irreversible. The equilibrium pressure above the oxide formed in the process is immeasurably small. Hydrogen sorption is reversible and the equilibrium with respect to outside is a function of the quantity of hydrogen dissolved in the metal. These conditions offer new possibilities regarding methods of gettering. Materials such as barium tend to remove all gases irrespective of their nature. While gases containing oxygen are obnoxious, hydrogen is not harmful and may even be beneficial in some cases. The use of thorium as a getter facilitates the effective removal of all oxygen and the retention of a residual hydrogen pressure at a predetermined value at the same time, acting as a permanent reducing atmosphere in a thermionic valve, for instance.

Sommaire : Etude détaillée des propriétés getter de poudre de thorium par rapport à l'hydrogène et l'oxygène.

Article by S. Wagener *Proc. Phys. Soc.* **66B**, May 1953 400-413

Glass Leak and Control Valves

136/I

United Kingdom. Three modifications of a valve design are described which was developed to control the flow of corrosive gases in evacuated systems. The valve is operated by differential expansion of either two different types of glass or two glass tubes of identical composition kept at different temperatures. The actual sealing element is a conical or a spherical joint, the male and female part of which are supported independently by the outer and inner tube respectively of a coaxially arranged pair of tubes. The outer tube made of borosilicate glass carries a Nichrome winding which can be heated electrically. If the inner tube is made of the same glass, it is water-cooled. But, if the inner tube is made of silica, no water cooling need be provided. The difference of axial expansion between the tubes causes the male member of the seal to withdraw from the female member. The size of the ' leak ' so formed may be regulated by adjusting the temperature difference. The rates of flow of air are shown in a graph, plotted against temperature up to 450°C. The pressure drop across the valves was 15 lb./inch². The shut-off rate of flow was found to be approximately 0.5 l.μ/sec.

Sommaire : Déscription de trois types de vannes fonctionnant selon le principe, qu'une fuite est produite en appliquant de la chaleur, resultant en une expansion différentielle à l'intérieur de la vanne.

Note by E. R. Harrison *J. Sci. Instrum.* **30**, May 1953 170-171

A Vacuum Valve to Provide Small Controlled Leak Rates

137/I

United States. A glass valve developed for use in the field of mass spectrometry or gas discharge research is described. It requires no vacuum grease for its operation and facilitates accurate control of gas flow rates over a wide range of leak rates. It consists of a thick-walled tube closed at one end and connected to the vessel to be pumped at the other. A jacket which is to hold the gas to be admitted to the system is sealed on to the tube. The centre portion of the jacket consists of a nickel sleeve, 1 inch long, which is joined to the glass *via* two Kovar cylinders. After mounting the jacket the assembly is cut cross-sectionally. The cut surfaces are optically polished and placed in contact with each other. After evacuation of the inner tube the two sections of the jacket are sealed. The nickel sleeve is surrounded by insulating material which carries a nichrome wire coil acting as the heater element. On heating the sleeve the jacket expands longitudinally and the inner abutting surfaces separate. In the closed position the valve has maintained a pressure of less than 0.01 micron Hg for one week in an 0.6 litre system. By heating the sleeve to any temperature below 200°C a predetermined and reproducible leak rate can be obtained which will remain steady for an indefinite period. Beyond 200°C nickel is likely to distort resulting in a deterioration of the sealing action of the valve.

Sommaire : Construction d'une vanne qui depend, pour son effet de fermeture, du degré de contact entre deux surfaces polies.

Note by R. Forman *Rev. Sci. Instrum.* **24**, April 1953 326-327

A Study on Diffusion Pump Oil by the Mass Spectrometer and the Gas Analysis of the Final Vacuum

138/I

See Abstract No. : 90/II

The Mass Spectrometer

139/I

United States. Basically the function of the mass spectrometer is to sort and identify atoms or molecules. The material investigated is ionised and the ions are passed through a magnetic field which forces them to travel on a circular path. The heavier the ion the larger will be the radius of the circular path and thus ions of different mass can be made to hit different targets. As the speed, the magnetic field strength and the travelling path is known the mass of the various ions can be recorded photographically or electrically. In the former case the instrument is called a mass spectrograph. For the correct working of the instrument it is essential to maintain a high vacuum in the instrument chamber in order to avoid that the ionised particles collide with the residual gas on their flight. The instrument owes its origin to the findings of J. J. Thomson in 1897. Working on gas discharges he found that cathode-rays were made up of particles, which had always the same mass and electrical charge (the discovery of the electron) and that the anode rays were positively

Vol 3 195

I — General Science and Engineering — I
Contd.

Abstract No. and References

charged ions, the mass of which differed and depended on the atomic weight of the gas present. He established the uses of a magnetic field for the separation of ions according to their weight. Using this method for the study of atomic weights he was able to prove that some elements have isotopes, *i.e.*, atoms of slightly different weight but the same chemical nature. In 1918 A. Dempster built the first mass spectrometer; in 1919 F. W. Aston constructed the first mass spectrograph. The latter used the instrument for a thorough study of isotopes. This work, together with Einstein's contribution, led to a method of calculating the latent energy in atomic nuclei, the discovery of uranium 235 and the use of the mass spectrometer for the production of that material for the atomic bomb. In a modern instrument the ions are produced by electrons emitted from a tungsten filament and accelerated through a potential difference of about 100 volt. to give the required energy for ionisation of the gas. The gas sample is introduced into the instrument chamber through a controlled leak, the chamber being pumped continuously. The ions are accelerated passing through a pair of slits in electrically charged metallic plates and travel on a straight path until they enter the magnetic field. After separation in that area they hit a predetermined target consisting of a plate. The impact causes a small electric current to flow which can be measured. The main industrial use of the mass spectrometer lies in the analytical field. It is applied in the petroleum industry for individual analysis or as a monitoring instrument, where it is difficult to analyse a mixture by ordinary chemical means. A remarkable installation of this kind is the monitoring system of the Oak Ridge U-235 gaseous diffusion plant where a series of instruments is used at various points, each instrument making 3,600 determinations a day automatically. In the same plant a portable version of the instrument is used as a leak detector to check for vacuum tightness at 1,000 test points. The instrument is used in biology for the analysis of respiratory gases under various conditions and in conjunction with stable isotopes in tracer experiments. Finally, its uses are described in geological investigations aiming at the determination of the age of the earth.

Article by A. O. C. Nier *Scient. Amer.* March 1953 68-74

Sommaire : Construction, fonctionnement et application du spectromètre de masse sont présentés.

Application of Vacuum Technique to the Production of Blood Plasma and Analogous Biological Products 140/I

See Abstract No. : 49/IV

Freeze Drying in Toxicology 141/I

See Abstract No. : 48/IV

The Sublimation of Ice in Vacuo 142/I

United Kingdom. The investigation reported here concentrates on the following principal cases : (*a*) sublimation of pure ice *in vacuo* and (*b*) sublimation of ice from a frozen solid *in vacuo*. In the section (*a*) it is stated that a solid sublimes if the partial pressure of its vapour in the system is below the triple point pressure. During the process heat is absorbed equal to the sum of the latent heat of fusion and evaporation. The rate of sublimation is described by the kinetic equation

$$G = \alpha (P_s - P) \sqrt{\frac{M}{2\pi R T_s}} \qquad (1)$$

where G is the observed rate of sublimation, α is the accommodation coefficient, P_s is the saturation vapour pressure of the ice, P is the condensation vapour pressure, M is the molecular weight, R the gas constant and T_s the temperature of the ice during sublimation. As the condenser is normally operated at a temperature very much lower than that of the subliming ice surface the maximum rate of sublimation can be expressed as

$$G = \alpha P_s \sqrt{\frac{M}{2\pi R T_s}} \qquad (2)$$

Sublimation can only proceed if heat is supplied to the subliming surface. Thus G can be expressed by

$$G = \frac{K_1}{\lambda} \cdot \frac{T}{x} \qquad (3)$$

where K_1 is the thermal conductivity in cal./cm.^{2} sec. °C per cm. thickness of the bed of ice, λ is the latent heat of sublimation in cal./gm. and T is the temperature difference (in °C) across a bed, x cm. thick. Further, in terms of heat input, H (cal./cm.^{2} sec.), the rate of sublimation is

$$G = \frac{H}{\lambda} \qquad (4)$$

In practice, for the sublimation of pure ice in vacuum, all but the first equation should be satisfied. Experiments to verify this were conducted in a drying chamber fitted with a special balance device for weighing the subliming ice in vacuum. This device permitted comparatively large amounts of ice to be used in the experiments. Details of the arrangement are given. The condenser was 7 cm. away from the ice surface and cooled with liquid oxygen. The temperature of the ice was measured by thermocouples at different depths. The heat input during any one experiment was kept constant. The sublimation rate and the surface temperature of the ice were found to remain constant during any one experiment. During the first stage of the experiment—evacuation—the temperature of the ice bed fell steadily. During the second stage—filling the

I — General Science and Engineering — I
Contd.

Abstract No. and References

condenser—the ice temperature dropped suddenly and the sublimation rate increased for a short while but decreased as the ice temperature continued to fall. At the third stage—switching-on the heater—the temperature at the bottom of the ice block rose to a maximum after which it fell progressively. Following these initial stages the ice surface temperature remained the same throughout and the temperature of the bottom face of the ice gradually approached that of the subliming surface. As shown in a graph the temperature gradient between top and bottom face of the ice remained substantially constant as sublimation proceeded at a constant rate. The outcome of the experiment proved that the three equations 2, 3 and 4 were satisfied. As the heat input was kept constant, the sublimation rate was to be constant (Equ. 4). Since the sublimation rate was constant, the temperature of the ice surface had to remain fixed (Equ. 2). And since the latter did not vary, the temperature gradient through the ice bed had also to remain constant (Equ. 3). The fact that the observed rate of sublimation was about half the theoretical maximum rate is attributed in part to the same conditions as those which account for the measured value of 0.54 for the accommodation coefficient, *i.e.*, the fact that the mean free path was less than the distance from the ice surface to the condenser in the experiment. In Section (*b*) it is stated that the presence of a solid complicates matters as, during sublimation, the ice recedes progressively into the material so that the path of the vapour through the solid lengthens steadily. Investigating the passage of water vapour through layers of clay experimentally it could be proved that the flow through the clay was of the Poiseuille type. Thus the rate of sublimation could be expressed by a simplified form of the Poiseuille equation :

$$G = \frac{P^2}{RL} \qquad (5)$$

where G is the mass rate of flow per unit cross section per unit time, P is the upstream pressure, L is the thickness of the dry so'id R is a factor indicating the ' resistance ' per unit thickness per unit area, depending on the nature of the solid. In the case of sublimation of ice from a solid Equ. 5 must be satisfied in addition to the equations developed in Section (*a*). Details are given of experiments to verify these stipulations using clay as the solid. From these experiments it was concluded that initially the sublimation of ice from a solid follows Equ. 1 but as the ice recedes into the solid the sublimation rate gradually assumes the values expressed by Equ. 5 due to the resistance of the dried solid. At this stage, as can be shown by equating Equ. 4 to Equ. 5, that the heat input must be progressively reduced if isothermal conditions are to be maintained.

Sommaire : Recherches théoriques et expérimentales sur la sublimation de la glace sous vide, telle qu'elle influe sur les méthodes de lyophilisation.

Article by E. W. Bruce *J. Imp. College. Chem. Engng. Soc* 6, 1950-1951-1952 39-51

Metal Evaporation in High Vacuum — 143/I

See Abstract No. : 98/III

The Coating of Plastics by Vacuum Evaporation — 144/I

See Abstract No. : 115/III

High Vacuum Metallising of Plastics — 145/I

See Abstract No. : 114/III

Good Carpets from Good Cops — 146/I

United States. A carpet mill, faced with the demand for higher output, installed a vacuum impregnation plant for the treatment of filling cops with liquid size replacing the conventional atmospheric process of dip-impregnation. The firm can now size 2,000 cops in less than one hour. Due to the more even distribution of the size in the pores of the yarn, the texture of the finished carpet is improved. The processing plant covers 800 sq. ft. and consists of a size cooking–and a size storing vessel operated at 190°F, a vacuum tank of 500 gallons capacity and a vacuum pump and condenser. 45 cops are bundled together and 15 bundles of cops placed on a tray. The vacuum tank holds three trays fully loaded. After charging, the tank is evacuated to a pressure of 29 inch. and kept at that pressure for 20 minutes. Then the hot liquid size is admitted to the tank. When all cops are submerged in the size the valves are closed and a pressure of 85 lb. per sq. inch applied for 30 minutes. Subsequently the excess size is drained and the cops are transferred to a humidity-controlled storage room.

Sommaire : Emploi du vide dans la fabrication de tapis : Les bobines sont imprégnées d'un liquide améliorant la texture du produit fini.

Article by Anon. *Textile Industr.* Jan. 1953

Paper for Punched Cards — 147/I

Germany. Punched cards with good dimensional stability and high strength are prepared from paper which is run under high vacuum through a vat containing synthetic resin (I). I includes alkyd resins, vinyl polymers, polystyrene, urea resins, etc.

(*Chemical Abstracts*)

Sommaire : Le traitement sous vide du papier pour des cartes (percées) est décrit.

F. H. Schroers & Schroers Jacquard Papier Industrie *Ger. Pat.* 808,668

I — General Science and Engineering — I
Contd.

Abstract No. and References

13 — ELECTRICAL SCIENCES — 13

An Electrical Analogue to a High Vacuum System — 148/I

United Kingdom. At low pressures (mean free path larger than the dimensions of the system) the rate of mass flow through a tube is given by the product of pressure difference and speed of tube, the latter depending only on its dimensions. Similarly, for a diffusion pump the mass flow is given by the product of pressure and speed at the throat. Hence, by comparison with Ohm's law, mass flow corresponds to the current in an analogous electrical network, if at the same time voltage corresponds to pressure and resistance to the reciprocal of speed. The use of an electrical analogue in the case of complicated vacuum systems, as for instance in betatrons and synchrotrons, eliminates tedious calculations of the effect of altering parts of the pumping system on pressure and shortens the time spent on leak detection as the answers can be provided quickly by simple voltage measurements. The electrical analogue of the Oxford 140–MeV synchrotron vacuum system is shown in a diagram and by way of example the procedure of locating leaks in this plant is explained in detail.

Letter by M. J. Aitken *Brit. J. Appl. Phys.* 4, June 1953 188

Sommaire : Analogie éléctrique employée pour faciliter l'estimation rapide des effects sur la pression occasionnés par la modification de différentes parties d'un système à vide complexe.

Combined Pirani and Ionisation Gauge Circuit — 149/I

See Abstract No. : 96/II

Work Functions of Gas-Coated Tungsten and Silver Surfaces — 150/I

See Abstract No. : 58/IV

Field Emission : Large Current Densities, Space Charge, and the Vacuum Arc — 151/I

See Abstract No. : 56/IV

Electrical Discharges and the Vacuum Physicist — 152/I

United Kingdom. This paper discusses briefly the various kinds of discharges which can occur in evacuated apparatus, *i.e.*, for ranges of pressure approximately equal or smaller than 10^{-4} mm. Hg when the mean free path is of the order of the dimensions of the apparatus. Under these conditions the processes of ionisation in the gas are not always the predominating processes of either initiation or maintenance of such discharges, which are then greatly dependent upon the nature of the electrode surfaces. The mechanisms of the various discharges which can be produced in this way are briefly discussed, and an indication given of the importance of the possible roles played by magnetic fields and by the nature of the electrode surfaces.

(*Author*)

Article by F. Llewellyn Jones *Vacuum* 3, April 1953 116-127

Sommaire : Discussion succincte des différents genres de décharge, pouvant se produire dans on appareil à vide.

Positive and Negative Ions in Cathode-Ray Tubes — 153/I

United States. The presence of positive and negative ions during operation is known to contribute to the failure of cathode-ray tubes. In previous investigations the negative ions have been identified and in a more recent publication (see *J. Appl. Phys.*, **22**, (1951), 1308) a double-ended hollow-cathode demountable apparatus was described, designed to obtain information on the positive ions. The present paper deals with the continuation of that investigation. It was found that the use of the apparatus mentioned presented difficulties due to gas evolution and, therefore, the work was carried on with sealed-off tubes. The tubes had conventional electro-statically focussed guns and the positive and negative ion burn spectra were separated by magnetic deflections. Examination of a large number of tubes showed that the most common ions were H_1^+, H_2^+, C^{+-}, O^{+-}, $C_2H_2^-$, CO^+. Ion currents of different components were measured in special tubes having a Faraday cage collector fixed to the tube face in an off-centre position. The magnetic field was adjusted to bring the desired ion component onto the collector slit. Measuring ion current intensities certain time effects were observed which appeared to be related to the degassing of components and apertures caused by the electron bombardment. Various special tube designs and treatments were investigated to determine and, if possible, to eliminate the source of ions. Thus graphite coatings were shown to contribute greatly to the carbon ion content. No significant differences were noticed in qualitative comparisons between tubes having tungsten filament or oxide cathodes, hydrogen or vacuum fired parts and tubes which had been pumped by mercury or oil pumps. Tests with half-aluminised tubes showed that ion burn was considerably reduced in the aluminised portion and that the remaining burns were due to penetration. The spot structure of negative ion burns was investigated. They originate mainly from the cathode and in some cases from apertures, positive ions were mostly generated by bombardment of surfaces by electrons. A discussion of the possibility that occluded and residual gases contribute to the generation of ions concludes the paper.

Article by H. Bachman, G. L. Hall & P. A. Silberg *J. Appl. Phys.* 24, April 1953 427-433

Sommaire : Certains aspects d'une recherche sur la source d'ions positifs et négatifs dans un tube à rayon cathodiques.

Vol. 3 1953

I — General Science and Engineering — I
Contd.

Abstract No. and References

A Demountable Flash-Discharge Tube for Production of the Lyman Continuum

154/I

United Kingdom. Three types of light sources are available to be used as backgrounds for absorption spectroscopy in the vacuum ultra-violet; the hydrogen continuum extending from the near ultra-violet to 1,650 Å, the Lyman continuum ranging from 1,650 to 900 Å and the Hopfield helium-2 continuum ranging from 900 to 600 Å. The Lyman continuum is produced by periodically discharging a condenser at a high potential through a narrow-bore quartz tube under conditions of extreme current density. The early designs of discharge tubes fitted with constrictions for the discharge path and mounted ' end-on ' to the spectrograph slit showed excessive wear of the capillary and a tendency of capillary debris to be thrown in the direction of the spectrograph slit. Worley developed a special design which eliminated both these drawbacks and provided for easy replacement of the capillary. In the present article a modification of Worley's design is described incorporating glass-to-metal seals and ' O '-rings in appropriate parts of the lamp in order to simplify servicing operations and to ensure accurate alignment of all internal parts on the spectrograph axis during assembly. A sketch of the design is shown. The main Pyrex envelope has a Kovar seal at the front and a ground flange at the rear. The Kovar seal is soldered to a brass flange which holds an ' O '-ring for attachment to the instrument. The flange at the rear is secured by an ' O '-ring and a collar to a large brass turning which carries the cathode supported by three steel rods. An inner tube holding the capillary and its shank made of Vitreosil in a packing of glass wool ends in a flange which is held between ' O '-rings against the large brass turning on one side and against a smaller brass turning on the other side, the latter carrying the aluminium anode. Normally the capillaries have a bore of 1 mm. In the present case the absorption vessel of the instrument was a King furnace separate from the spectrograph and the light source had to be kept 40 cm. away from the slit. Therefore a capillary with an initial diameter of 4.5 mm. is employed. This widens to 6.5 mm. after some 1,500 flashes. With the discharge tube in this position some 100 flashes are adequate to produce satisfactory blackening of the plate. Due to the larger-size capillary a larger capacity condenser bank has to be employed. Details of the electrical equipment are given.

Sommaire : Construction améliorée d'un tube à decharge (flash) développé par Worley, pour la production du Spectre de Lyman entre 900 et 1,650 Å.

Article by W. R. S. Garton *J. Sci. Instrum.* **30,** April 1953 119-120

Electrodeless Metal-Halide Lamps

155/I

United States. Meggers and Westfall used electrodeless discharge lamps to obtain spectra of mercury. The present authors report on the production and use of similar lamps in order to obtain spectra of less volatile metals. The absence of sealed-in electrodes reduces the evolution of gases and the danger of contamination of the spectrum. Compared with open arcs sharper lines are obtained showing less self-absorption. The metal to be examined is introduced into the lamp in the form of a halide which melts between 200° and 500°C. The discharge, initiated and maintained in argon or helium present in the lamp at a pressure of a few mm. Hg, causes the halide to vaporise and dissociate, exciting the atomic spectrum of the metal in the process. There is no need to use a volatile salt, if the metal to be examined has a vapour pressure of approximately 1 mm. Hg at the operational temperature. The lamp is made from Pyrex tubing for studies in the visible range of the spectrum and from Vycor for studies in the ultra-violet. One end of the tube is sealed. A few mg. of the salt are introduced and the other end is connected to a pump. During evacuation the upper end of the tube is heated red hot for degassing while the lower end containing the salt is protected by a wrapping of wet asbestos paper. Later, the paper is moved to the upper part of the tube, the salt deposited by distillation on the cool upper wall and the lower part of the tube is degassed. Then the gas is admitted and the lamp is sealed off. In this manner, lamps charged with halide salts reasonably stable in moist air, such as $CuCl_2$, $FeCl_3$, $NiCl_2$ and UCl_4, have been constructed in 1 to 2 hours. Where the halide salts are subject to severe hydrolysis they cannot be obtained commercially and must be specially prepared. Details are given of the manufacture of a beryllium lamp by using beryllium bromide or alternatively beryllium iodide as an initial material where the salt is formed in the lamp itself after evacuation. The lamps are operated in an electric field of a frequency greater than 100 Mc. A suitable power plant with 100 watt maximum power output is mentioned. The discharge is started with a high frequency leak tester. The spectral lines of the halogen and the noble gas can be easily identified by comparison with a halogen and rare gas lamp. The molecular bands appear weakly or not at all and can be avoided altogether by the choice of a suitable halide. The spectra are free from self-reversal, but some self-absorption is evident.

Sommaire : L'on décrit la production de lampes à vapeur sans électrodes utilisées pour la génération de spectres de métaux difficiles à volatiliser.

Article by C. H. Corliss, W. R. Bozman & F. O. Westfall *J. Opt. Soc. Amer.* **43,** May 1953 398-400

The Preparation of Titanium Dioxide Beam-Splitters of Large Surface Area

156/I

See Abstract No. : 106/III

I — General Science and Engineering — I
Contd.

Abstract No. and References

14 — MECHANICAL SCIENCES — 14

HF Brazes High-Vacuum Joints 157/I

United States. The construction of magnetrons (u.h.f. radio wave generators) provides for the joining of a variety of metals, often in close proximity of the glass envelope. The production procedure adopted in a particular case is described where 17 separate joints are required. Nine are brazed in a hydrogen furnace and eight are brazed with high frequency current. The latter consist of 5 joints on the cathode assembly itself and 3 seal-in brazes. The forming gas in the high frequency process is a 10/90% mixture of hydrogen and nitrogen in the first case and pure hydrogen in the second. The fixtures required for the high frequency current method are all similar in design. They are of the bench type, vertically and laterally adjustable to ensure proper location of the work in the h.f. coil. The enclosures are metal frames fitted with panels of clear plastic and carry an explosion door at the top. A single turn work coil is used to produce the required surface heat in the work. The electrical energy is supplied at 400,000 c./sec. from a generator *via* a transformer at low voltage. Each generator serves four transformers, interlocked in such a manner that only one operable position is available at any one time. The first cathode assembly braze joins a molybdenum cone to a cathode sleeve. Wetting and adequate heat conduction of the brazing surface is ensured by facing the molybdenum cone with sintered nickel powder. As the operational pressure of the magnetron is 5×10^{-6} mm. Hg oxygen-free, high conductivity copper brazing wire is used and assembled with the parts to be brazed. Its melting point is 1,083°C. The cone only is inductively heated. The heat reaches the wire and the sleeve by conduction. The brazing temperature is held until wetting is complete ensuring a vacuum tight seal. The assembly is cooled below red heat in the forming gas and then to less than 100°C in pure nitrogen. The second joint is made between a thin Kovar sleeve carrying a glass envelope assembly and the upper end of the molybdenum cathode sleeve. The third h.f. braze joins a solid nickel heater to a Kovar sleeve at the opposite end of the glass envelope, using a 0.020 inch. diameter copper-silver eutectic ring. This solder has no plastic state at its melting point (779°C), flows quickly and facilitates the completion of the operation before heat can affect the glass section. The fourth braze joins the copper- and nickel-plated cathode support cylinder to the molybdenum cone and the final braze joins a nickel-plated Kovar ring on the cathode envelope to the support cylinder. To eliminate oxidation the assembly is evacuated in this operation before admitting the forming gas. The article concludes with a detailed description of the brazing operations required in joining sub-assemblies such as the window cup cathode and tuning head to the main body assembly.

Article by H. W. Garbe *Machinist* 16.5.53 799-802

Sommaire : Details d'une méthode de brazage de joints à vide poussé à l'aide de courants d'Eddy.

15 — PHYSICS — 15

The Sublimation of Ice in Vacuo 158/I

See Abstract No. : 142/I

Techniques for the Electron Microscopy of Crystals 159/I

See Abstract No. : 104/II

The Effect of Vapour Incidence on the Structure of Evaporated Aluminium Films 160/I

United Kingdom. Evaporated aluminium films deposited on polished glass at a thickness of 0.05 to 0.2 micron give specular reflection. If the thickness increases above 1 micron the film surface becomes coarse, and scatters incident light. The same conditions can be obtained at smaller film thickness by directing the vapour beam at a high angle of incidence to the substrate. This effect was first observed in experiments concerned with the coating of glass cylinders. Using an extended vapour source parallel to the axis of the cylinder and rotating the cylinder during the process it could be shown that the granulated structure of the deposit became increasingly pronounced as the distance between the source and the cylinder decreased. With the cylinder stationary a specular band was produced in the area of normal incidence with diffused reflecting strips on either side. The specular band narrowed as the deposit grew thicker. This led to the belief that the onset of diffused reflection depends on the deposition of a critical mass of aluminium at any given angle of incidence. Methodical experiments were then conducted to determine the critical surface densities which produce diffused reflection. Details of the experimental technique are given. The operational pressure was 10^{-4} mm. Hg. The evaporation temperature was restricted to 1,150°C. Under these conditions collisions between atoms in the vapour beam take place only in the immediate neighbourhood of the filament and truly spherical distribution of the vapour and an accurate angle of incidence can be expected. The source-surface distance was 24 cm. and the incidence angle could be measured by special means within $\pm 2°$. The light source was a tungsten lamp operated at constant voltage and the reflectivity was measured with a photo-multiplier tube combined with Wratten filters No. 50 and 24 A giving maximum sensitivity of 4,600 and 6,000 Å respectively. Films deposited at an angle of vapour incidence not exceeding 70° showed full reflectivity when opaque but the specular reflectivity fell as the surface density increased. The surface densities of films

Vol. 3 1953

I — General Science and Engineering — I

Contd.

Abstract No. and References

condensed at various angles in that range were determined at the stage, where their reflectivity at 4,600 Å was approximately 4% less than the 90% normally obtained from aluminium films, and plotted *versus* the angle of incidence. The curve becomes asymptotic near normal incidence indicating that a smooth and compact structure could be obtained even with thick films if the vapour rays would form a strictly parallel beam. But at higher angles of incidence increase of surface density resulted in a granular surface giving a whitish matt appearance producing specular reflection only if the light was directed to the film at a glancing angle. This indicated the presence of elevated particles on the film surface which could be eliminated by burnishing the film surface with cotton wool. After this treatment specular reflection could be obtained from all angles. Films deposited at angles of vapour incidence exceeding 70° showed scattered reflection of light before the deposit became opaque. Their absorption is higher and their reflectivity is lower than that of films deposited at normal incidence indicating agglomerated structure. From the observations made in the experiments it is concluded that initially the atoms forming the condensation nuclei are evenly dispersed on the substrate surface but as long as the vapour atoms continue to arrive at an angle the nuclei grow edgewise. When they have reached a certain size, the vapour atoms arriving will no longer penetrate the interspace between the grains and in the final stage grain and interstices will grow in the direction of the vapour beam. Thus, as experimentally observed an obliquely deposited film shows high transmission for light travelling in a direction parallel to the incident vapour beam.

Article by L. Holland *J. Opt. Soc. Amer.* **43**, May 1953 376-380

Sommaire : Rapport sur des expériences méthodiques en vue d'obtenir des informations sur l'effet d'angle d'incidence de vapeurs sur la structure des couches minces déposées, en utilisant l'aluminium comme matérial d'essai.

Study of the Crystalline Grain in Thin Films of Gold or Palladium or Gold-Palladium Alloys Prepared by the Thermal Evaporation Technique. 161/I

France. Specimens of thin evaporated metal films have been examined in the electron diffraction camera by the transmission technique and the average size of the crystals has been determined from the diameter of the rings observed by a method described in detail. Three factors influence the crystal size : The thickness of the film, the properties of the metal and the nature of the substrate. The condition of the substrate remained the same throughout the experiments but the thickness of the films was varied in the range below 350 Å. At this or higher values it is no longer possible to determine the ring diameters accurately. The results for gold are shown in a table reproduced below.

Thickness of the film (Å)	1.5	5	20	200
Size of the Rings (mm.)	1.7	1.4	0.8	0.4
Average Size of the Crystal (Å)	15	20	35	80

Palladium films varying in thickness from 5 to 80 Å show an average crystal size of approximately 25 Å changing by no more than 10% over the whole range of thicknesses. Alloys of the two metals containing 60% gold content have cubic face centred crystals typical of compound crystals. The size of the crystals is approximately 25 Å, the same as in the case of pure palladium. The crystal size does not change when the gold content is reduced to 30% gold. The outcome of the experiments proves that shadow casting with a gold-palladium alloy will give a better resolution than shadow casting with pure gold.

Note by L. Lafourcade *C. R. Acad. Sci., Paris* **236**, 8.6.53 2220-2221

Sommaire : La grosseur des cristaux d'or, de palladium ou d'un alliage des deux, en forme de couche mince, evaporée sous vide, a été determinée par la technique de la diffraction électronique.

The Preparation of Titanium Dioxide Beam-Splitters of Large Surface Area 162/I

See Abstract No. : 106/III

On the PbS Photoconductive Cell 162/I

Japan. In the past thermopiles and bolometers have been used as infra-red detectors. But for the particular purpose of the authors the time constant of the conventional instruments was not satisfactory. Bose described in 1901 a PbS photoconductive cell of high sensitivity and small time constant in the near infra-red and the authors describe the construction and performance of a cell based on Bose's design. The cell consisted of a glass tube 4 cm. long and 1 cm. diameter. Two tungsten wires were sealed-in at one end and brought into contract with two strips of aquadag applied to the interior of the tube wall serving as the electrodes. While the tube was pumped 2–3 mg. of PbS Powder were evaporated with the help of a gas flame. This process was repeated until the material was properly deposited in the area forming the gap between the two aquadag electrodes. Before finally pumping down to 10^{-4} mm. Hg and sealing off the tube was heat-treated in order to raise its conductivity. Maximum conductivity was obtained at 380°C. To ensure optimum sensitivity oxygen was leaked into the tube during evaporation to remove harmful reaction products such as SO_2. Besides PbS, galena and PbS_2O_3 were tried out but PbS was found to be the most satisfactory material. The electrical equipment employed in testing the cell is described in detail. The amplifier system used consisted of a pre-amplifier representing a modification of the Ritter circuit, connected to a two-stage R.C. coupling amplifier. The illumination could be interrupted at any frequency between 50 and 2,000 cycles by a mechanical chopper.

I — General Science and Engineering — I
Contd.

Abstract No. and References

The dark resistance of the cell varied from 0.1 MΩ to 20 MΩ. The response of the cell to illumination intensity was linear up to 10 μ W/cm.^{2}. This was satisfactory for the requirements of the authors. The sensitivity of the best cell in the series was 1.9×10^{-10} watt at room temperature when exposed to the light of a lamp with a colour temperature of 2,355°K and a radiation density of 8.3 μ W/cm.^{2} at 1 m. distance. The time constant was found to be 2.8×10^{-4} seconds at 15.5°C. Details of the method employed for the measurement of the spectral response of the cell are given and the results are shown in a graph. The maximum response is in the red at 0.6 μ wavelength and the second peak is at 2.3 μ. The PbS cell was found to be appreciably more sensitive than a thermo-junction in the near infra-red. The cell was used in an infra-red spectrometer fitted with a glass prism instead of a rock salt prism. As a result the slit could be made narrower facilitating higher resolution. Spectrograms taken with the help of the PbS cell and, alternatively, the thermopile showing the spectra of liquid benzene and the absorption bands of water and carbon dioxide contained in atmospheric air are given.

Sommaire : Détails d'un détecteur infrarouge en forme de cellule photoélectrique en PbS développé en vue d'un temps de réponse court.

Article by N. Sassa & M. Nakamura *Sci. Light* **2**, No. 2, 1953 104-115

Multilayer Dielectric Reflecting Films at Wavelengths between 4,000 and 4,600 Å. 164/I

○ *United Kingdom.* Due to lack of data available it is not possible to assess the relative merits of dielectric and silver coatings for Fabry-Perot interferometer plates at wavelengths below 5,200 Å. The authors prepared multilayer dielectric interference filters on glass in the form of a pair of seven quarter-wavelength layers made from 4 zinc sulphide and 3 cryolite films, the two composite films being separated by a half-wavelength cryolite spacing layer. The individual layers were deposited in the form of a wedge so that the wavelength λ giving a first-order pass band varied over the filter throughout the range considered. The half intensity width and peak transmission of a number of these filters was measured in the range of 4,000 to 4,600 Å and the values for N (ratio of the wavelength of the pass band to its half width) and τ (the fraction of the incident light transmitted at the peak of the pass-band) were plotted in a graph. From this the mean values of reflection, transmission and absorption for the pair of films in each filter could be determined. At 4,500 Å the values were : reflection coefficient = 0.983, transmission coefficient = 0.014 and absorption coefficient = 0.003. In theory if air was substituted for the spacing layer the reflection coefficient would be about 0.005 higher but the ratio of transmission to absorption would remain constant. N is directly proportional to the resolution obtainable. Thus silver and dielectric films can be compared as follows : for $\tau = 0.45$ at 4,200 Å silver films give N = 16 whereas dielectric films give N = 250, *i.e.*, the resolution of the latter at this wavelength is 15 times higher than that of the former. At 4,600 Å the dielectric filters give a half-width of 22 Å and a peak transmission of 70%. The corresponding figures for silver are: A half-width of 150 Å and a transmission of 40%. Irregularities of the glass support have no effect on the performance of multilayer dielectric filters.

Sommaire : Les avantages d'un film dielectrique multiple dans la région en dessous de 5,000 Å ont été étudiés et comparés à des films d'argent du type utilisé dans les étalons Fabry-Perot.

Letter by J. Ring & W. L. Wilcock *Nature* **171**, 11.4.53 648-649

On the Choice of Measurements to be Made for Simultaneous Determination of the Optical Constants and Thickness of Thin Absorbing Films 165/I

See Abstract No. : 109/III

○ **The Optical Constants of Gold and Silver in the Ultra-Violet** 166/I

Germany. The constants are determined by the method of Kretzmann (1940) using four reflections from evaporated films. For silver, thickness = 180 mμ, λ = 330–550 mμ. For gold, thickness = 350 mμ, λ = 250–550 mμ.

(*B.S.I.R.A. Bulletin*)

Sommaire : Les constantes de l'or et de l'argent ont été étudiées pour l'ultra-violet à l'aide de films évaporés.

Article by H. Kober *Ann. Phys., Lpz.* **11**, No. 1, 1952 12-14

Improvement of Grating Efficiency in the Vacuum Ultra-Violet by Platinising 167/I

United States. During work described in Abstract 99/I unexpectedly high spectral intensities were recorded below 1,500 Å during measurements in a monochromator. This effect was traced to be due to the unintentional coating of the grating with platinum sputtered from the light source. To investigate the matter further a rather poor grating with deep grooves was sputtered intentionally with platinium under vacuum. The first coating was 400 Å thick and no improvement could be noticed but when the thickness reached 1,600 Å the efficiency increased appreciably. The amount of light scattered by the grating was not changed, and the resolution not affected. A table, partly reproduced below, shows the numerical value of the factor R by which the grating efficiency was increased by platinum coating.

Vol. 3 1953

I — General Science and Engineering — I

Contd.

Abstract No. and References

Wavelength (Å)	1,650	1,500	1,350	1,100
R for the grating coated unintentionally	0.8	1.5	2.5	25
R for the grating coated intentionally	1.0	7	20	—

Sommaire : Il a été constaté que le recouvrement non intensionnel d'un réseau, pendant les mesures d'intensité à l'ultra-violet, avec du platine de la source lumineuse, a augmenté le rendement du réseau. Des essais plus poussés du phénomène sont rapportés ici.

Letter by K. Watanabe *J. Opt. Soc. Amer.* **43**, April 1953 318

A Demountable Flash-Discharge Tube for Production of the Lyman Continuum 168/I

See Abstract No. : 154/I

16 — CHEMISTRY — 16

New Field for High Vacuums 169/I

United States. Manufacturers of heavy chemicals begin to take an interest in high vacuum concentration. Caustic soda can be concentrated to a 35% solution in vacuum at a temperature not exceeding 150°F. Magnesium chloride concentrated in vacuum is not hot enough to liberate corrosive HCl gas. The rate, at which the plant material corrodes, depends on the temperature and velocity of the processed material. Tests on mild steel in a salt solution show that a reduction of the temperature from 60 to 20°C reduces the corrosion rate from 0.045 cm. p.a. to 0.01 cm. p.a. A reduction of the velocity from 9 to 3 ft. p.sec. reduces the corrosion rate by 25%. In the particular process described the velocity is low because only the force of gravity acts on the liquor to be concentrated. Low-temperature operation retards the formation of scale on the heat-exchange surface. Black liquor was concentrated from 10 to 50% solids at a temperature of 100°C for 60 hours, at the end of which the heat-transfer capacity had only decreased slightly. The plant described consists essentially of a falling-film evaporator. The heat for evaporation is supplied by water, warmed up to 90°F by condensing Freon 11 contained in a refrigeration unit, which supplies water at 37°F to a barometric condenser for the condensation of the vapours coming from the evaporator. The operational pressure of the unit is about 10 mm. Hg produced by a three-stage steam jet ejector backing up the barometric condenser.

Sommaire : Il a été trouvé avantageux de concentrer des matériaux corrosifs tels que la soude caustique, sous vide, car le degré de corrosion est fortement réduit, ce qui résulte dans des économies de construction et d'utilisation, des appareillages.

Article by Anon. *Chem. Engng.* June 1953 134-136

Potash Refining in France 170/I

See Abstract No. : 133/III

A New Apparatus for the Determination of Moisture Content 171/I

United Kingdom. The checking of materials for moisture content is an important test in the chemical and many other industries. The methods ordinarily employed are either volumetric or gravimetric. The former, also known as distillation methods, are quicker than the latter, but have the disadvantage that more or less inflammable solvents are required for the operation and the apparatus has to be carefully cleaned before each test. Any traces of water present will distort the results. An apparatus has been designed which is simple to operate and reduces the time required for the test appreciably. It consists of a cylindrical glass vessel covered with a dome-shaped glass lid serving as the drying chamber. An electric hot plate is mounted inside the chamber which is supplied *via* a lead introduced into the chamber through a rubber stopper, sealing an opening in the wall of the chamber. The same stopper carries an air-inlet nozzle. Another stopper sealing an opening in the lid carries a thermometer and a glass tube fitted with a stopcock serving as the connection to a water-jet pump. With this apparatus the water content of a rosin emulsion with a bone-dry content of 25% was determined in 10-15 minutes.

Sommaire : Détails d'un nouvel appareil pour la détermination de la teneur en humidité.

Article by G. Kurz *Paper-Maker* Midsummer 1953 14

Freeze Drying in Toxicology 172/I

See Abstract No. : 48/IV

Vol 3 195

I — General Science and Engineering — I
Contd.

Abstract No. and References

Application of Vacuum Technique to the Production of Blood Plasma and Analogous Biological Products 173/I

See Abstract No. : 49/IV

An Improved Historadiographic Apparatus 174/I

See Abstract No. : 99/II

Storage of Normal Tissue Grafts 175/I

United Kingdom. A Symposium on the ' Preservation of Normal Tissues for Transplantation ', organised by the Ciba Foundation, was held in March 1953. Various speakers dealt with the scope and limitation of freezing and freeze drying techniques in this connection as follows :— *J. W. Pate.* Certain homografts need not be alive on transplantation and the complications involved in storing in the living state should be avoided where possible. Freeze-dried dead arterial homografts are preferable to fresh living homografts and freeze-dried bone is as good as living bone. Freeze-dried skin has been used successfully as a temporary dressing on severe burns. *P. L. Mollison.* The preservation period of red blood cells can be effectively prolonged by adding glycerol to the storage media which facilitates the application of lower temperatures. *C. M. Pomerat.* During investigations aimed at ascertaining the relationship between bulk behaviour of tissue and response of the individual cells, apparatus has been developed which record thermally and photographically the behaviour of cultivated cells when exposed to thermal stress. *R. E. Billingham.* Skin slowly frozen to –79°C and impregnated with a glycerol solution did not show any deterioration after storage for 10 months. Glycerol has negligible toxic effects. Freeze drying of skin is satisfactory only, if the residual moisture content is not less than 25%. *F. K. Sanders.* Freeze-dried nerve homografts to bridge a gap in the nerve trunks were successful in the case of small laboratory animals but not in the case of human beings. This may be due to the fact that the gaps to be dealt with in normal clinical practice are larger than in laboratory animals. *G. C. Rob and H. H. G. Eastcott.* Arterial homografts used after storage in the frozen state have given good results independent of the length of the graft and its viability at transplantation. The new endothelial lining was apparently formed by cells derived from the blood stream. *C. A. Hufnagel.* Experiments with vascular grafts on dogs have proved that the success of a graft is independent of whether it was fresh, frozen or freeze-dried. Freeze drying, no less than any other method of treatment, requires that the grafts are preserved in an aseptic solution. Ethylene dioxide is suggested for the purpose. *W. R. Strong.* The work of the U.S. Naval Tissue Bank was described. Skin is stored after freeze drying to less than 1% residual moisture and used as temporary dressing. It eases pain, controls the loss of tissue fluid and counteracts infection. Freeze-dried bone has given encouraging clinical results.

Sommaire : Rapport sur une réunion couvrant des conferences présentées au symposium de ' Preservation de Tissus Normals par Transplantation ' organisé par la fondation Ciba en Mars 1953.

Article by Anon. *Nature* **171**, 13.6.53 1047-49

Direct Determination of Oxygen in Rubber 176/I

United States. In the past oxygen has been determined in rubber by difference in weight, but this method is slow and, lately, direct analytical methods have been developed. For instance, the Schuetze-Unterzaucher method can be employed for the purpose. The rubber sample is vaporised at 1,000°C and carried over pure carbon, heated at 1,150°C in a stream of nitrogen. The oxygen content of the sample forms carbon monoxide which is converted into carbon dioxide over iodine pentoxide heated to 120°C. The iodine liberated in the process is determined quantitatively and serves as an indication of the oxygen content of the rubber sample. In 1946 an isotopic method was developed which permits the determination of oxygen in organic compounds without requiring separation of the oxygen. The isotopic method is based on the isotopic dilution principle first introduced by von Heresy and Paneth and described in detail by Grosse *et al.* (see *J. Amer. Chem. Soc.*, **68** (1946), 2119). The rubber samples are placed in a platinum boat. The boat is inserted into a platinum combustion tube and the tube is sealed to a vacuum system. Combustion of the rubber with oxygen–18 enriched oxygen gas admitted to the tube takes place at a temperature of about 900°C, which is maintained for 30–60 minutes. Various gases are formed in the process including carbon monoxide and dioxide. At this temperature level all the oxygen atoms exchange statistically with the gaseous oxygen-containing compounds and the stable residue but the ratio of the oxygen isotopes in all these compounds is identical. It is sufficient, therefore, to determine the ratio of oxygen–18 to oxygen–16 in one of these gases, preferably in carbon dioxide. In order to isolate the carbon dioxide the system is evacuated *via* a liquid nitrogen trap which retains the condensable gases. Then, the trap is replaced by a melting isobutane bath with a temperature of –145°C and pumping continued at a pressure not lower than 1–2 mm. Hg leaving behind highly concentrated carbon dioxide since the vapour pressure of carbon dioxide at that temperature is 0.24 mm. Hg. The ratio of oxygen–18 to oxygen–16 in the carbon dioxide is determined in a mass spectrometer. When this ratio is known, the oxygen content of the sample can be calculated by a formula given in the article. A table, partly reproduced below, shows the results obtained with the isotopic and the Unterzaucher method using samples of the same rubber.

Vol. 3 1953

I — General Science and Engineering — I

Contd.

Abstract No. and References

Table I. Determination of Oxygen Content of Smoked Sheet Rubber by Isotopic Method and Unterzaucher Method

Sample	State of Oxidation	By Isotopic Method: Wt. of Sample, mg.	Wt. of Oxygen-18 Gas, mg.	Atom % O^{18} above Normal Concentration in Equilibrated Mixture: in b	Atom % O^{18} above Normal Concentration in Equilibrated Mixture: Observed % Oxygen	By Unterzaucher Method, Observed % Oxygen	Difference, Av. % Oxygen (I — U)	% Oxygen, Calculated by 'Gain in Weight': Gain	% Oxygen, Calculated by 'Gain in Weight': Total
1	Unoxidised	418.62	5.50	5.1080	1.7932	2.46	1.20		0 2.50
		409.77	6.69	5.0795	2.0657	2.42	1.38		
		424.24	6.35	5.0795	1.8606	2.59	—		
		418.42	6.89	5.0795	1.9973	2.55	—		
						Av.2.50 ±0.07	1.29 ±0.09	1.21	
3	30 hours in O^2 bomb at 70°C. Oxidation 2 at Lee Rubber & Tyre Co.	351.54	7.04	5.1080	1.7932	2.27	0.85		
		352.78	4.99	5.0795	1.8357	2.50	0.93		0 2.38
		328.34	4.23	5.0795	1.8287	2.36	—		
						Av.2.38 ±0.08	0.89 ±0.04	1.49	
5	218 hours in O^2 bomb at 70°C. (Sample 3 oxidised for additional 188 hours)	314.60	6.99	5.0795	1.6433	4.66	3.42		2.48 4.86
		415.73	7.39	5.0795	1.3892	4.82	3.80		
						Av.4.74 ±0.08	3.61 ±0.19	1.13	

a % Oxygen by ' Gain in Weight ' calculated by adding the weight gain upon heating O_2 to the values obtained by isotopic method for samples 1 and 3.
b No gain in weight obtained.

The results obtained by the latter method are invariably lower than those of the former. In order to come to a proper assessment independent calculations were made based on ' gain in weight '. The calculated values are very close to those obtained by the isotopic method but those of the Unterzaucher method are persistently lower, up to 62% in terms of actual oxygen values. The Unterzaucher method depends for its accuracy on the complete vaporisation of the sample, but rubber does not vaporise completely, and in a special experiment it was proved to contain oxygen which is not accounted for in that method.

Sommaire : Les mérites d'une méthode Isotopique nouvellement développée pour la détermination directe de l'oxygène dans le caoutchouc, sont comparés à ceux de la méthode Schütze-Unterzaucher, pour le même but.

Article by A. D. Kirshenbaum & A. G. Streng *Analyt. Chem.* **25**, April 1953 638-640

Adsorption by Evaporated Copper Films at 78°K. Part 1. Krypton and Hydrogen 177/I

See Abstract No. : 40/IV

Adsorption by Evaporated Copper Films at 78°K. Part 2. Krypton and Oxygen 178/I

See Abstract No. : 41/IV

Kramer and Russell Effects with Single Crystals of Zinc 179/I

United Kingdom. Kramer showed that upraded metal surfaces emit negatively charged particles which activate an open-ended Geiger counter operated in air. Whereas Kramer assumed that the particles in question were electrons emitted as a result of phase changes on the surface, the authors found that the particles were oxygen-molecule ions. Furthermore it appeared that the ' active ' areas created by the surface treatment have a lower work function than is usually attributed to the particular metal. This condition was confirmed in experiments with single crystals of zinc cleaved in the (0001) plane. One cleaved surface was upraded, the other was not. The two surfaces were investigated with an open-ended counter operating at 10 cm. Hg pressure and exposed to daylight through glass cutting off the ultra-violet wavelengths. The upraded surface gave 275 counts per minute and the other surface gave 40 counts per minute. The photoelectric response of zinc is normally cut off at 3,000 Å. In this case it was cut off at 5,000 Å. In another experiment the Russell effect was used. A zinc crystal treated as before was exposed to a photographic plate for 20 hours. The upraded surface gave a dark image but the other surface gave hardly any image at all.

Sommaire : Le mécanisme de phenomènes connus comme effets Kramers and Russell a été étudié avec du zinc comme matériel d'essai.

Letter by L. Grunberg & K. H. R. Wright *Nature* **171**, 16.5.53 890

I — General Science and Engineering — I

Contd.

Abstract No. and References

17 — METALLURGY — 17

Research into Metals 180/I

Germany. Recent progress in the development of research tools for the study of metals is surveyed. In the past investigations of the structure depended essentially on the resolving power of the light microscope. The introduction of the electron microscope improved the resolving power available to 20 Å. Examinations in the electron microscope are carried out by the transmission technique. For this reason, a replica of the specimen to be examined is normally required. Various examples of modern replica techniques are given. In the case of thin films, replication is unnecessary. Another new laboratory tool is the electron diffraction camera which, as distinct from the more conventional X-ray diffraction method, facilitates the investigation of the surface structure of extremely thin films, such as evaporated films and fired layers of metal. Knowledge of the mechanism of reactions such as the progress of diffusion processes becomes increasingly important from the point of view of surface finish quality. The sketch of an arrangement is shown which facilitates the continuous recording of the kinetics of such processes using the electron diffraction method. A record obtained in this manner, of the progressive changes of the crystalline structure of Ag_2S, while heated gradually to 180°, is given. In order to permit quick checks on production conditions a special instrument has been developed which consists of a light microscope fitted with a hot plate. A sample of the processed material is placed on the hot plate and heated up to a pre-determined temperature either in vacuum or in an inert gas atmosphere. The instrument can be used for the investigation of diffusion phenomena as shown by the following example. A piece of tantalum sheet was etched with sulphuric acid and subsequently covered with ' Eloxyl ' (an oxygen-rich solution) so that bluish green interference colours appeared uniformly all over the surface. Heating the tantalum on the vacuum hot plate the oxygen diffused slowly into the interior of the material and the colour of the sheet changed. The speed of the diffusion was governed by the orientation of the polished grains in the surface. At 560°C the crystals which originally appeared dark had changed to red and violet and the crystals which originally had a bright appearance had changed to a bluish green or bluish violet. These colour changes continued until at 690°C the whole surface had assumed a uniform bluish colour indicating that all oxygen of the solution had been taken up by the surface and diffused into the interior. In another application of the instrument a piece of a platinum crucible employed for the heat treatment of metals was heated up under vacuum on the hot plate. Observation in the microscope established that phosphorus had been taken up by the platinum as a result of faulty production procedure. The author proceeds to discuss the production of pure metals with particular reference to the vacuum melting of beryllium, titanium, zirconium and tantalum. A laboratory induction heated vacuum furnace for processing at 2,000°C and pressures of 10^{-4} mm. Hg is shown in an illustration. It follows a brief discussion of the research on thin films.

Sommaire : Revue des téchniques modernes employées pour l'étude des propriétés des métaux en masse ou couches minces.

Article by A. Boettcher *Aus Forschung und Producktion* 1953 7-31

A Versatile Vacuum Melting Furnace 181/I

See Abstract No. : 124/III

Vacuum Furnaces for Melting or Processing of Metals 182/I

See Abstract No. : 121/III

Electrical High-Temperature Furnaces, Fitted With Molybdenum Resistance Heaters 183/I

Germany. The service life of resistance heaters made from alloys and evaporated in industrial furnaces is governed by the melting point of the processed material and the design of the furnace. The maximum operational temperature of chromium-nickel alloys is 1,100° and of chromium-iron-aluminium alloys 1,200°C. Globar (silicon carbide) can be heated up to 1,350°C. At higher temperatures platinum, molybdenum or tungsten has to be used. Molybdenum is cheaper than the others. It can be easily machined and has a fine grain structure after heat treatment. Its melting point is 2,650°C but in industrial furnaces its operational temperature does not exceed 1,800°C. At high temperature the permissible load is 15 to 20 watt/cm², as compared with 0.8 watt/cm² for chromium-iron-aluminium alloys. Molybdenum at 1,600°C has a resistivity about 9½ times that of cold molybdenum. For this reason the heater must be protected electrically, when switched on. In atmosphere the molybdenum heater can only be heated to 400°C. At higher temperatures molybdenum must be operated under a reducing atmosphere or in a vacuum. The protective gas must suit not only the heater but also the processed material. Hydrogen supplied in cylinders is normally used. Hydrogen is highly inflammable, its ignition temperature lies between 560° and 620°C, depending on the mixture with air. Hydrogen is expensive. Where large quantities are required it should not be burnt off but a hydrogen circulation plant should be provided. Means of eliminating oxygen and moisture from the gas are discussed. Another suitable protective gas can be obtained by splitting ammonia (NH_3) into hydrogen (=75%) and nitrogen (=25%). One kg. of liquid ammonia yields 2.8m³ of dissociated gas. Dissociated ammonia is absolutely dry and can be used directly in the heat treatment of iron and steel, brass, bronze, tungsten and stainless steel. A table is shown giving operational data of molybdenum reduction and sintering furnaces ranging in size from 65 × 50 × 800 mm. to 350 × 100 × 1,500 mm. Forging, hardening and soldering ovens for operational temperatures up to 1,650°C are described. A vacuum is provided in the furnace where the melt has to be degassed in order to improve the

Vol. 3 1953

I — General Science and Engineering — I

Contd.

Abstract No. and References

density and elasticity of the finished product, also in cases where the processed materials are sensitive to the protective gases and finally in order to eliminate impurities, the vapour pressures of which are higher than those of the processed material. There has been a great demand recently for alloys of rare metals such as copper-zirconium, copper-titanium, nickel-zirconium and nickel-titanium. These materials are highly sensitive to oxygen and must be produced in high vacuum. Materials for permanent magnets, processed in vacuum, show an increase in permeability and a decrease of hysteresis losses. Distillation of metals in vacuum eliminates volatile impurities. In this way selenium, tellurium and sulphur are removed from electrolytic copper. There are many processes which require higher temperatures than are available in a molybdenum furnace. In that case high frequency furnaces should be used but the molybdenum furnace covers the needs of a very large section of industry.

Sommaire : Discussion détaillée des fours à resistance de molybdène couvrant tous aspects de leurs performances.

Article by F. Regel *Planseeber. Pulvermet.* 1, June 1953 97-116

Determining Gas in Aluminium Melts

184/I

United Kingdom. It is well known that hydrogen present in the molten metal is the cause of unsound castings but not all melts are required to be completely gas-free. An intermediate hydrogen content prevents undue concentrations of shrinkage unsoundness at changes in section but it is difficult to control the actual amount left in the melt. A quick and reliable method of estimating the gas content is needed in the foundry. A reduced pressure test for a qualitative assessment by comparison has been described recently (see Abstract No. 58/I). A quantitative assessment can be provided by the determination of the density of a DTD test bar casting, but this method is slow. A quicker method has now been published in the British Patent No. 684,865. It is based on the established relationship $G=K\sqrt{P}$, where G is the gas content and P the gas pressure in equilibrium with the molten aluminium at constant temperature. Thus, if the solubility of hydrogen in aluminium at a given temperature is S_o at 760 mm. Hg pressure, the equilibrium pressure P_1 in mm. Hg corresponding to another gas content G_1 is given by:—

$$G_1=S_o\sqrt{\frac{P_1}{760}}$$

The method requires the measurement of the pressure developed inside a closed palladium tube, permeable to hydrogen and inserted into the melt. The upper end of the palladium tube is brazed to a closed platinum tube element situated in an impervious ceramic tube the latter being attached to a hood of the same material. A steel protected copper tube conveys the hydrogen which has penetrated from the melt into the measuring system to a mercury manometer. The copper tube connects also *via* taps to a vacuum reservoir, and an argon supply. In operation argon is admitted to the system in order to eliminate any oxide film which may be present on the surface of the melt. Then the system is evacuated by connecting it momentarily with the vacuum reservoir. Finally the system is isolated and the equilibrium pressure read on the manometer.

Sommaire : La détermination de teneur en gaz, des bains de fusions, par la méthode de Ransley, qui facilite une détermination rapide, est décrite en détail.

Article by Anon. *Light Metals* 16, April 1953 130.

Electrodeless Metal-Halide Lamps

185/I

See Abstract No. : 155/I

19 — SCIENCES (other than the above) — 19

Apparatus for Vacuum Firing of Porcelain Dental Articles

186/I

United States. A dental laboratory device for custom firing of porcelain jackets and bridges, etc. at low pressure is described. It has been noticed that vacuum-fired components of this kind show a greater brilliance and translucency in various colours including those of the natural teeth. Also, due to the absence of air-pockets and bubbles the material is particularly dense and harder then the components fired by conventional means. The device consists of a bench furnace housing a firing chamber positioned in the centre of the furnace base. The firing chamber is appreciably smaller than the furnace chamber in order to create an evacuated space around it assisting in the reduction of heat losses. The open end of the firing chamber can be blocked. In front of the firing chamber but within the furnace chamber is a small platform to store temporarily objects to be fired. Heating is carried out electrically and is controlled by rheostats. The heater elements are embedded in the walls of the firing chamber. The maximum operational temperature is 2,500°F. A vacuum pump is connected to the furnace. A valve is fitted to control the admission of atmospheric air when required. A pyrometer and a vacuum gauge are placed in suitable positions.

Sommaire : Petit four à vide de table utilisé pour cuire des dentiers en porcelaine.

N. Gold & Nu-Dent Porcelain Studios Inc. *U.S. Pat.* 2,636,915

II — Vacuum Apparatus and Auxiliaries — II

Abstract No. and References

20 — PUMPS — 20

Methods of Obtaining High Vacuum by Ionisation. Construction of an 'Electronic Pump' 85/II

Brazil. The first suggestion of an ion pump came from Plucker in 1858 who proposed to make use of the electrical clean-up mechanism for the purpose. But the absorbing parts of such a pumping device would soon become saturated and in order to ensure that the pumping action is continuous the ions of the gas pumped must be drawn to a region where they are neutralised and pumped away by a backing pump. The number of ions formed (n_i) must always be greater than the number of gas molecules ($\triangle N$) which diffuse back into the receptacle pumped, *i.e.*, $n_i > \triangle N$. This condition is applied to the case of a simple discharge tube x cm. long, 2r cm. wide, fitted with a cathode at one end and an anode at the other, and both sides of the inequality are calculated. n_i depends on the number of electrons emitted from the cathode, on the difference of potential between the electrodes, on differential ionisation (the number of ions reduced to 1 mm. Hg obtained for each electron that collides with gas molecules at least once on its path), the pressure and the length of the electron path x. $\triangle N$ is governed by the Knudsen law for the case, where the mean free path is longer than the diameter of the tube. The average pressure is assumed to be 10^{-4} mm. Hg and the ultimate pressure to be 10^{-6} mm. Hg. The results of the calculation are shown in a table, partly reproduced below. As can be seen the length of the tube required is reasonable at large electron currents but a large space charge will develop

Minimum Length of Discharge Tube Suitable for Pumping

Ie (*amp.*)	*Xmin* (*cm.*)	*Ii* *ion current* (*amp.*)	*S* *at* 10^{-4} *mm. Hg* (*l./sec.*)
For r = 1.0 cm.			
0.001	735	1.4×10^{-4}	0.13
0.1	118	8.5×10^{-4}	0.8
1.0	53	1.9×10^{-3}	1.8
10.0	24	4.2×10^{-3}	4.0

in operation which, it is calculated, will require an electrical field of the order of 10^5 volt to ensure the movement of the electrons and for that reason a simple d.c. gas discharge is not a practical solution. It is important that the path of the electrons is lengthened without increasing the length of the tube. The electrons can be forced either to oscillate about the axis of the discharge tube or to move in spiral paths applying a magnetic field. Suitable construction data can then be obtained from calculations as follows :–r = 1 cm.; ν (increase of the length of the electron path) = 20; $I_e = 0.1$ amp.; $x_{min.} = 16$ cm. S = 6 l./sec. at $I_e = 0.2$ amp. and $p = 10^{-4}$ mm. Hg. The ion current in this example is about 5 mA. Therefore no space charge develops. The author describes an ion pump developed in 1940. It consisted of a tube fitted with a hot cathode at one end, a ring-shaped anode at the other and a third annular electrode of the same potential as the cathode. The electron current was 10 mA and was kept oscillating through the annular anode at about 2,000 volt. A magnetic field parallel to the tube axis ensured movement of the electrons on a spiral path. As this design showed extensive clean-up a new model was developed with four openings in the glass wall between two ring electrodes in the centre and one opening at either end of the tube. Five of these openings were connected to a common rotary pump and the sixth was connected to the receptacle to be pumped. In front of each opening was a ring-shaped electrode, which was at a negative potential in the case of the five openings connected to the pump and at a positive potential in the case of the sixth, to prevent ions from diffusing back into the receptacle. A volume of 500 c.c. could be evacuated from 10^{-3} to 10^{-5} mm. Hg in less than one second with this pump.

Sommaire : Recherche théorique sur les possibilités d'obtenir de véritables effets de pompage par des procédés d'ionisation. Description d'une construction pratique d'une pompe à ionisation basée sur ces considérations.

Article by H. Schwarz *Rev. Sci. Instrum.* **24**, May 1953 371-374

Improvements i.o.r.t. High Vacuum Diffusion Pumps 86/II

United States. The design of an oil diffusion pump is described which has been specially developed to facilitate short pumping cycles in such cases as the pumping of radio-valves and which will tolerate the admission of atmospheric air for short periods while it is hot. The cylindrical pump body contains a lower annular jet system and an upper annular jet system. The former rests on a tube of a diameter appreciably smaller than that of the pump body and the latter on a tube of still smaller diameter. The pump body is fitted with cooling coils in the conventional manner. The pump body and lower jet tube rest on the boiler, the diameter of which is appreciably larger than the pump body. At the lower end of the annular space formed by the lower jet tube and the pump body, a wire coil is provided tack-welded to the pump body. The wire diameter is about the same as the distance between the two tubes. The presence of the coil will (*a*) ensure accurate centering of the jet assembly, (*b*) prevent vapours rising from the boiler reaching the pump chamber, (*c*) provide a long path for the condensed oil returning to the boiler and (*d*) by virtue of the presence of the condensed oil act as a protective vapour seal, if the pump is exposed to a sudden inrush of air. As the air which has penetrated into the pump is dealt with by the backing pump, it will be removed through the jets and not *via* the boiler portion of the pump minimising entrainment of oil in the process.

Sommaire : Description d'une pompe à diffusion à système déflecteur annullaire.

R. L. Lawrance, J. L. Middleton & National Research Corp. *Brit. Pat.* 684,470

Vol. 3 1953

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

Improvements i.o.r.t. Condensers for Oil Diffusion or Vapour Jet Vacuum Pumps

87/II

Liechtenstein. Baffle plates or condensers, fitted to the mouth of an oil diffusion pump, are used to prevent back diffusion of vapours. In order to be suitable the condenser must have a temperature just above room temperature and must be so designed that no obstruction is offered to the movement of the air molecules pumped. The particular design described consists of a number of flat rings of different diameters spaced apart along their common axis and forming a frustro-conical structure opening out towards the vessel to be pumped. The rings are held in position by four bolts screwed into a cup-shaped body at the apex of the frustro-conical structure which fits the cap of the pump jet system and is water-cooled to stop the heat radiation from the jet cap and to keep the rings cool (by conduction). The oil condensed on the rings flows along the bolts to the cup-shaped body and from there over an inclined sheet to the wall of the pump. All parts of the condenser should be made from copper, chromium plated, where necessary to avoid decomposition of the oil by catalytic action of the copper.

Sommaire : Dessin d'un baffle à place entre pompe à diffusion et enceinte à évacuer.

A. Vogt
Brit. Pat. 691,434

Vacuum Pumping Apparatus

88/II

United States. An ejector type of oil vapour high vacuum pump is described which incorporates special means of returning the condensed oil to the pump boiler against the pressure in the boiler without depending on a hydrostatic head of the condensate. The oil condensed in the paired ejector and diffuser element which effects the pumping action, is returned to the boiler through a tube which is fitted with another jet and diffuser element pointing downwards, the jet being supplied with vapour from the boiler through a separate conduit of small diameter. The respective diffuser element is of divergent shape at its lower end which causes the velocity of the mixture passing through the diffuser to decrease and its pressure to increase. The vapour operating the second jet serves to pre-heat the mixture on its way to the boiler.

Sommaire : Construction d'une pompe à vapeur d'huile du type éjecteur.

J. H. Black, H. G. Warren & Eastman Kodak Co.
Brit. Pat. 683,275

High Vacuum Device

89/II

United States. Oil diffusion pumps employed as booster pumps normally have blank-off pressures of about 10^{-5}–10^{-4} mm. Hg and are required to operate against fore pressures of the order of 1 mm. Hg. The volumetric pumping speed of these pumps, if fitted with an upper and a lower jet, largely depends on the design of the lower jet. Due to the high fore pressure the gap between the lower jet umbrella and the inner wall of the pump body must be small to prevent back leakage, but if it is too small the flow of the gas pumped by the upper jet is restricted. In practice the width of the gap is determined by the thickness of the oil condensed on the wall of the pump body and returning to the boiler in the form of a falling film. The thickness of the film is governed by the viscosity of the oil and the viscosity of the oil depends on its temperature. It is proposed in this patent to ensure high pumping speeds by keeping the viscosity of the liquid oil low in the regions of the pump where this is important by providing adequate control of the cooling conditions. The conventional cooling coil is used. The coil is wound around the pump body from the top down to a level somewhat below that of the upper jet and is then connected by a straight section to the coil wound around the fore line of the pump which joins the main pump body just below the bottom jet. From here another straight section of the tube connects with the coil surrounding the upper part of the boiler and extending upwards in a helical winding to a level just above that of the lower jet. A valve is fitted to the inlet at the top of the coil to regulate the flow of the cooling water. In the arrangement described maximum cooling is provided in the area below the upper jet and in the fore line. Subsequently the water is heated up circulating around the boiler section and heats the oil flowing down the walls in the region between boiler and lower jet. In the case of a 4-inch. diffusion pump the gap between lower jet umbrella and wall should preferably be 0.15 inch. Using pentachlorinated diphenyl as the pump fluid in such a pump the temperature of the water should be 40–70°F at the inlet and 77–95°F at the outlet. At the latter temperature the viscosity of the condensed oil should be less than 50,000 Saybolt Universal seconds and its vapour pressure below 10 micron Hg, ensuring rapid flow of the oil in a thin film of a thickness not exceeding 30% of the gap width and avoiding back-streaming of oil vapours.

Sommaire : Description d'un dispositif spécial pour pompes booster facilitant le réglage approprié du refroidissement le long des parois de la pompe pour assurer un écoulement rapide du film d'huile condensé à l'épaisseur correcte vers le bouilleur.

R. B. Lawrance & National Research Corp.
U.S. Pat. 2,630,266

A Study on Diffusion Pump Oil by the Mass Spectrometer and the Gas Analysis of the Final Vacuum

90/II

Japan. In order to obtain information on the composition of the residual gases in a system pumped by an oil diffusion pump it is necessary to have full knowledge of the composition of the pump fluid used. The authors selected for their experiment a 98.5% pure alkyl-naphthalene oil, supplied by the Lion Oil and Fat Co. Ltd. which has a low vapour pressure and great stability. Its main constituent is claimed to be hexadecyl-naphthalene. The oil was analysed in a mass spectrometer constructed in the Tokyo Institute of Technology The operational data of the instrument is given. The oil employed in the pump of the instrument was the same as the material under examination. A liquid air trap was fitted to the spectrometer pump. In order to obtain a complete spectrum of the oil, scanning had to be carried out repeatedly at varying magnetic field strength. As the peaks of the same ion varied with changing accelerating voltage a correction factor had to be applied which had been worked out beforehand in comparative measurements with argon gas. Three small containers

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

which could be sealed off separately were attached to the ion source. They contained alkyl-naphthalene, D.O.P. and mercury respectively. The latter was used to produce the mercury ions Hg_{202}^{+} and Hg_{202}^{++} which were adopted as standards. The operational pressure in the ion source was 10^{-6} mm. Hg and the temperature was 150°C. The collecting current was kept below 40 μA as it was found that the ion yield was linear in that range. The spectrogram obtained is shown in an illustration. Its values indicate that electron impact causes alkyl-naphthalene to decompose into various molecular ions of lower mass number. Before the spectrum of the oil was recorded a blank test was run with the liquid air trap out of action. This test facilitated the identification of the residual gases in the instrument chamber by comparison with the spectra obtained for the oil in the manner described before. The composition of the residual gases in approximate proportions is given in a table reproduced below. No evidence could be found of thermal decomposition of the pump fluid. Blears'

Table III

Mass Number	*Material*	*Percentage*	
2	H	1.0	1%
17 }	H_2O	6.5 }	20%
18 }		14.5 }	
28	$CO+N_2+CO_2$	4.4 }	6%
44	CO_2	1.2 }	
32	O_2	1.1	1%
	Alkyl-Naphthalene	59.0	59%
	the Others	13.0	13%

claim that a tungsten filament operated in an evacuated system causes the formation of carbon monoxide in the presence of water vapour and hydrocarbons has been confirmed by the authors in an experiment measuring the ion peak of mass number 28, with the ionisation gauge, connected to the instrument, switched on and off at regular intervals. The peak was reduced to 50% some 5 seconds after the gauge had been switched off.

Article by K. Histake & K. Matsuda *J. Phys. Soc., Japan* 8, May-June 1953 416-421

Sommaire : Le spectromètre de masse a été utilisé pour analyser l'atmosphère résiduelle dans la chambre évacueé muni d'une pompe à diffusion fonctionnant avec une huile de marque connue.

Supersonic Speeds in High Vacuum

91/II

Germany. Thermodynamic considerations led to the conclusion that the speed of the vapour emerging from the jets of diffusion pumps is supersonic. A device is described which facilitates visual demonstration and photographic recording of the flow conditions in front and at the back of the jet. It consists of a boiler holding mercury connected by a tube to a jet which is interchangeable. The jet discharges into a tube of transparent material which returns the mercury *via* a condenser to the boiler. Two electrodes are inserted in the tube at the back of the jet and another pair of electrodes is inserted in front of the jet. A glow discharge between the electrodes maintained at a suitable high tension varies in intensity as the pressure varies. The experiments described were conducted in a range of pressures from 10^{-1}–10^{-4} mm. Hg. At the higher pressures accurately defined flow pictures were obtained which agreed well with ' Schlieren ' photographs of the respective characteristic flow conditions. By placing an adjustable wire sonde in the path of the vapour stream it is possible to measure the flow speed by means of the Mach angle. Mach numbers up to 2.5 have been measured in this way. The method described is recommended for the testing of jets in high vacuum.

Note by K. Klumb, E. Robens & O. Scholz *Naturwiss.* 40, June 1953 196-197

Sommaire : Description d'un dispositif qui permet de rendre visibles les conditions d'écoulement sous vide, lequel pourrait être utilisé pour les essais des déflecteurs sous vide.

21 — GAUGES — 21

Improvements i.o.r.t. Two Stage Liquid-Ring Air Pumps

92/II

Germany. The first stage of a two-stage liquid ring pump for the production of a high vacuum is preferably designed for a greater displacement than the second as the amount of gas exhausted to obtain a high vacuum is larger than the quantities delivered by the second stage operating at a moderate vacuum. The efficiency of such a pump deteriorates when it is operated at a low vacuum because the first stage delivers more gas to the second than the second can handle. To overcome congestion of this kind it is proposed to let the second stage run free of sealing liquid, *i.e.*, in effect, to by-pass this stage completely. The sealing liquid in the pump is constantly renewed by liquid drawn in from outside by the vacuum existing in the pump. The higher the vacuum the more liquid is drawn in by the first stage and passed to the second stage. According to the invention an auxiliary evacuation port is provided in the second stage which connects with the pressure side. The amount of liquid discharged through this port depends only on the size of the port and its location but is independent of the vacuum existing in the pump. Pumping at high vacuum sufficient liquid will reach the second stage to effect sealing in spite of the losses *via* the auxiliary evacuation port; but during pumping at a low vacuum the liquid present will not suffice to seal the second stage.

O. Siemen & J. Hinsch *Brit. Pat.* 691,425

Sommaire : Détails d'une construction récente d'une pompe à air à anneau liquide, à deux étages, destinée à éliminer la détérioration du rendement de la pompe par vide insuffisant.

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

The Limiting Negative Pressure of Mercury in Pyrex Glass

93/II

United States. The convex meniscus formed by mercury filled into a tube is generally considered proof that mercury does not wet glass. But Wichers *et al.* have found that thoroughly cleaned mercury filled into a clean evacuated ampoule forms a flat meniscus, confirming that there is adhesion between mercury and glass under certain conditions. Users of the McLeod gauge will know the phenomenon of the ' sticking vacuum ' in the measuring capillary, *i.e.* on release the mercury sticks to the capillary and develops a certain negative pressure before it is freed. The author conducted methodic experiments in order to ascertain the maximum negative pressure mercury could withstand. A pyrex-glass U-tube manometer sealed at one end was evacuated, thoroughly degassed and mercury distilled into it, while in a horizontal position. The length of the mercury column from the sealed end to the meniscus was 52 cm. The manometer was then mounted vertically and the open leg evacuated. It was found that the mercury column would not move and assume the same level in both legs until the tube was jarred. The negative pressure sustained by the mercury in this experiment was 2/3rd of an atmosphere. Further experiments were carried out using a centrifugal method. Short evacuated mercury thermometers were cemented to the horizontal spinner in a position where the end of the bulb was slightly further away from the axis of rotation than the meniscus. Eight thermometers were used. The negative pressures measured ranged from 2 to 17 bars (1 bar=0.986 atm.). These variations appeared to be due to differences in cleanliness and dryness of the thermometer capillaries. Therefore, another series of experiments was carried out using fine Pyrex capillaries, 20 cm. long which were evacuated, carefully degassed by heating and filled under vacuum. The results of the centrifugal tests for these capillaries are given in a table partly reproduced below. The highest negative pressure = 425 bars (at 27°C) shown in the table was obtained

Limiting Negative Pressure of Mercury in Pyrex Glass

Tube No.	*Pn (Bars)*	*Treatment*
9	46	Torched
11	7	Not Torched
14	338	Furnace, 450°–500°C
15	425	Furnace, yield point

after the centrifugal stress had been increased in 16 steps starting with a negative pressure of 120 bars. It is believed to be the highest negative pressure, measured directly for any liquid. The next highest is 318 bars measured for chloroform.

Sommaire : Il est prouvé que le mercure mouille le verre, sous certaines conditions, et développe une pression négative connue sous le nom d'effet collant, comme dans les jauge de McCleod.

Article by L. J. Briggs *J. Appl. Phys.* **24**, April 1953 488-490

On the Theory of the Quartz Fibre Manometer

94/II

Germany. Friction of the gases, normally independent of pressure, becomes proportional to the pressure in the low pressure range. Langmuir suggested to use this phenomenon as a means of measuring low pressure and thus initiated the development of the quartz fibre manometer. In its simple form a single fibre is provided which is suspended at one end. The gradual reduction of the amplitude of the swinging fibre is a measure of the pressure and is observed through a microscope. An alternative design provides for two fibres joined at their free ends forming the apex of a triangle. In this arrangement the fibres oscillate in a plane which is always normal to that of their suspension. The authors report on a theoretical investigation into the working principle of these instruments. In the case of the single-fibre manometer it has been assumed:–1. The oscillations of the fibre are of the nature of harmonic torsion oscillations of a fibre which is theoretically rigid. 2. The motion of the fibre has no effect on the Maxwell-Boltzmann velocity distribution of the molecules hitting the fibre. 3. The molecules are reflected elastically by the fibre. 4. The velocity of the swinging fibre is small compared with the thermal velocity of the gas molecules. The motion of the fibre is expressed by $J\varphi=-k\varphi+D$, where φ is the angle of the amplitude of the fibre oscillations, J the moment of inertia with respect to the pivoting point, k the directional moment and D the damping moment. D consists of two components $=D^*-D_0\varphi$ where D_0 is the self-damping factor and D^* the damping produced by gas friction. The latter is pressure dependent. Its value has been calculated by Haber and Kerschbaum but their results do not entirely agree with values found empirically. A stricter method of calculation taking gas-kinetic considerations into account yields results which agree with those published by Born. The expression arrived at by this method is

$$D^*=-\frac{C}{3}\sqrt{\frac{M}{RT}}\,r.L^3.p.\varphi$$

where 2r is the fibre thickness, L the total length of the fibre and $C=2\sqrt{2\pi}$ according to the strict theory. By writing the first equation in the form $\varphi+2\delta\varphi+w^2\varphi=0$ the damping constant can be expressed as $\delta=\frac{C}{\pi}\sqrt{\frac{M}{RT}}\cdot\frac{P}{\rho d}+\delta_0$ where δ_0 is the constant of self-damping. In practice reading a quartz fibre manometer one determines δ. The amplitude of the fibre oslillations diminishes exponentially with the time according to $A=A_0e^{-\delta t}$. Normally the time required to reach half the maximum amplitude only is measured which is related to δ by $t_2=1n\ 2/\delta$ Taking into account the previous formula $t_2=\frac{\text{const}\cdot d.}{\sqrt{M}.p+a}$ The value of the

Vol 3 195

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

constant calculated by the strict method is 112. In order to prove the validity of the theory the calibration curve of a double-fibre manometer was determined for air in the pressure range 10^{-5} to 5×10^{-2} mm. Hg. Above the pressure of 5×10^{-2} mm. Hg damping is smaller than assumed in the investigation, *i.e.* the assumption that the motion of the fibre has no effect on the velocity distribution of the molecules no longer holds good. In the particular case the calibration constant was found to be 128, a value deviating from theory. The values of calibration constants determined by other workers are shown in the following table. The values obtained for single-fibre

Constant of Various Quartz Fibre Manometers

Type of Manometer	*Author*	*Constant*	*Average Constant*
Single Fibre Manometer	Haber and Kerschbaum	115 113[a] 100[a] 110.5 113[a] 110[a] 114[a]	111
Double Fibre Manometer	Wetterer	114 129 158 122 163	139
	Huthsteiner	157	
	Mielenz and Schoenheit	128	

manometers are generally smaller than those obtained for double fibre manometers. This may be due to the fact that the joint between the two fibres is somewhat thicker than the single fibres. As is shown the theory of the single-fibre manomater cannot be applied to the double-fibre manometer without some qualifications but the operation of the double-fibre manometer is subject to the same laws except for minor deviations.

Sommaire : Recherche théorique sur le principe de fonctionnement du manomètre à fibre de quartz.

Article by K. D. Mielenz & E. Schoenheit *Z. Angew. Phys.* 5, March 1953 90-94

A Small Pirani Gauge for Measurements of Non-Steady Low Pressures

95/II

United States. For special work a gauge of small size and volume was required to operate in the pressure range of 0.1–10 mm. Hg. The report describes the design and performance of a Pirani gauge constructed of a matched pair of surgical grain-of-wheat lamps (11/32 inch long 0.082 inch outside diameter) mounted in a small metal block. Each lamp had a single loop of tungsten filament (0.00071 inch diameter) with a resistance of between 1.7 to 2.3 ohm at 72°F. One lamp was open to the pressure to be measured, the other was sealed at atmospheric pressure to give partial temperature compensation. The lamps were used in a constant-current Wheatstone bridge network, the out-of-balance current being a measure of the pressure. With the help of a programme device each bridge was switched momentarily for the pressure measuring circuit to a configuration, where half the bridge acted as a resistance thermometer to compensate for changes in the temperature of the gauge wall. Three pressure ranges were provided by a network in series with the recording galvanometer (140 μA F.S.D.). The power supply for operating 12 such gauges in parallel consisted of 6 wet-cell aircraft batteries giving 144 volt output, housed in a special room. The current for each Pirani gauge bridge was kept constant at 78.4 mA by a swamping resistance of 1,800 ohm. Provision was made for checking the current during each pressure measurement. The instrument was calibrated against an inclined tube butylphthalate manometer on a special calibration set covering the range 0–20 mm. Hg. The calculation for temperature correction is given. The performance is discussed in detail; time lags in indication were found to be less than 1 second. Errors in pressure indication did not exceed 2.5%. A theoretical analysis of the operation of the gauge concludes the report.

Sommaire : Détails du principe de fonctionnement d'une petite jauge Pirani pour utilisation dans le domaine compris entre 0.1 et 10 mm. Hg.

Report by M. J. Pilny *N.A.C.A. Rep. No.* TN2946 June 1953

Combined Pirani and Ionisation Gauge Circuit

96/II

Sweden. The Pirani gauge is of the direct-reading constant-temperature type developed by von Ubisch and the author. The Pirani element is a 0.01 mm. diameter tungsten wire of 80Ω resistance and forms one arm of a Wheatstone bridge circuit. This is connected as a feedback network to an audio-amplifier tuned to a frequency of about 1 kc/s by a phase shifting network so that the circuit will oscillate and keep the bridge near balance corresponding to a constant temperature of about 70°C above room temperature. The voltage across the bridge is a measure of the pressure covering the two ranges 10^{-3}–10^{-1} mm. Hg and 10^{-1}–50 mm. Hg. A relay circuit actuated by the gauge may be used to protect diffusion pumps and other equipment in case of vacuum failure. The sensitivity of the gauge to hydrogen is very much different than for air even at high pressures and this feature is valuable for leak detection at high pressures. The ionisation gauge consists of an emission

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

regulating circuit and a d.c. amplifier. The method of emission stabilisation is based in part on a circuit developed by Steckelmacher and Van der Meer (see Vol. 1, Abstract No. 17/IV). The entire ionisation gauge filament power is controlled by the regulating valves in a feedback circuit in which the ionisation gauge grid is compared with a reference voltage. The difference with the earlier ionisation gauge circuit is the use of two thyratron valves (2D21) in place of high vacuum output valves as regulating valves and the omission of the pre-amplifier. An emission stabilisation of 3% is obtained. A conventional balanced vacuum tube voltmeter is used to measure the pressure. Range and outgassing circuits are included using a single multi-range switch. A common stabilised d.c. power supply is used for both the Pirani and the ionisation gauge.

Sommaire : Description d'un circuit combiné de Pirani et à ionisation.

Article by G. von Dardel *J. Sci. Instrum.* **30**, April 1953 114-117

Improvements i.o.r.t. Electric Discharge Devices for the Measurement of High Vacuums 97/II

Germany. The design of a cold cathode ionisation gauge is described which, it is claimed, permits accurate operation of the gauge at pressures below 1×10^{-6} mm. Hg. Also, an a.c. supply can be used instead of the d.c. employed in previous designs. The instrument consists essentially of a pot magnet, the two poles of which serve as electrodes. One of the two poles is holed in the centre to provide a connection between the discharge chamber within the magnet and the receptacle, the pressure of which is to be determined. An additional annular electrode is situated in the instrument chamber half-way between the two other electrodes. As a result an entirely symmetrical discharge space is created facilitating a stable gas discharge even if a.c. is used. By covering the poles of the magnet with tantalum, harmful cathode sputtering effects can be minimised as the sputtered tantalum will combine with the residual oxygen present to form non-conducting tantalum pentoxide. In order to ensure an adequate supply of ions at very low pressures the tops of the electrodes should be coated with a radio-active preparation. The use of a pot magnet as described ensures a strong magnetic field and a minimum of magnetic leakage losses.

Sommaire : Un circuit combiné d'un jauge de Pirani et d'un jauge à ionisation est décrit.

M. Auwaerter *Brit. Pat.* 692,232

Improvements r.t. Pressure-Gauges 98/II

France. An aneroid capsule gauge is described which represents a simplification of the conventional construction and simple means of adjustment as is frequently required after exposure to vibrations. The pressure fluid is admitted along the axis and below the capsule. A drain valve is provided to remove condensate if necessary. The driving member of the mechanism moves vertically and contacts a lamellar spring positioned horizontally and fixed at one end. At the other end the spring carries a roller which is fixed to one end of an arm pivoting around a fixed point at its centre. The opposite end of this arm has the shape of a toothed quadrant meshing a pinion on the indicating needle. In order to counteract wear of the spring a nut carrying a slotted disc which supports the spring is mounted on a threaded rod in a position parallel and close to the spring. Normally the nut is near the fixing point of the spring but by rotating the threaded rod it can be moved in the direction of the free end of the spring when an adjustment of the resillience of the spring is required.

Sommaire : Détails d'une jauge à anéroide.

Aerocessoires Simmonds *Brit. Pat.* 668,488

22 — MEASURING PLANT (VACUUM) — 22

An Improved Historadiographic Apparatus 99/II

United States. The term historadiography covers the study of animal tissues by radiographic techniques and facilitates the determination of the mass of biological structures which may be as small as 1 micron in diameter. Reference is made to Goby who used the method for the first time in 1913, Lamarque who described a suitable apparatus in 1936 and Engstrom who published a theoretical discussion on the subject. The authors describe in great detail an apparatus of their own design which incorporates a number of improvements over previous designs, developed in the light of Engstrom's experiences:—Exposure time is reduced to 30-40 seconds, the pumping arrangements facilitate taking 10 radiographs in one hour and the timing of the exposure is accurately controlled. The essential features of the apparatus are :—A continuously evacuated demountable X-ray tube, a special vacuum interlock in the photographic chamber, an X-ray current stabiliser and an automatic timing circuit. The power is supplied by a rectifier unit which has an input of 115 volt with a capacity of 15 kVA and rectified output of 15 kV at 0.5 A. The stabiliser design is of the type employed by LeMieux and Beeman. Evacuation is carried out by an oil diffusion pump coupled with a rotary pump. The latter has a capacity of 102 l./min. and the former a capacity of 250 l./sec. at the pressure of 3×10^{-5} mm. Hg. A line by-passing the diffusion pump connects the fore pump with the outer portion of the photographic chamber. The presence of the interlock in the photographic chamber facilitates continuous running of the pumps. Thus, a vacuum of 10^{-4} to 10^{-5} mm. Hg is reached in tube and chamber within a few minutes of introducing the camera into the outer portion of the photographic chamber. The tube is separated from the photographic chamber by a 8μ aluminium foil in order to absorb the cathode light and filter the beam to give maximum intensity at 8-12Å. Details of the electronic timing circuit are shown in diagrams. The method of operation is fully described. The specimen and a reference of nitrocellulose are placed on a small disc separated by a slit. The preparation is put in direct contact with Lippman film and exposed at 3 kV and 30 mA. The mass of the specimen is determined by comparing the X-ray absorption of the specimen structure with that of the reference sample. The resolving power is limited by the grain size of the film used. Lippman film resolves approximately 1,000

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

lines per mm. The radiographs can be enlarged photographically 500 x. Several examples of the application of the method are quoted. For instance, the mass of various cells in fundic stomach was estimated with the help of this method where the magnitudes are of the order of 5×10^{-13} g.

Sommaire : Description détaillée d'une construction spéciale d'appareil à rayon-X pour la détérmination de la masse de structures biologiques pouvant être aussi petites qu'un micron en diamètre.

Article by J. J. Clemmons & M. H. Aprison *Rev. Sci. Instrum.* **24,** June 1953 444-454

The Mass Spectrometer 100/II

See Abstract No. : 139/I

High Temperature Mass Spectrometer Oven 101/II

United States. Several heating methods are known for the production of ions from solids for study in the mass spectrometer. The indirect method of heating affords good temperature control but the maximum temperatures obtainable are too low to effect vaporisation in many instances. Direct heating methods are more suitable. Reference is made in this connection to the designs of Bleakney, Blewett, Sherr and Smoluchowski, Sampson and Bleakney, and Palmer and Aitken. In the present report a cylindrical oven is described specially developed for the vaporisation of low vapour-pressure materials which are to be analysed in an all-metal 60° mass spectrometer with a Nier-type source. No thermal insulating material is used. The heating element consists of thin tantalum or platinum sheet (2 mil) cut into narrow strips about 60 mil wide from which cylinders are formed by drawing the strips through a number of wire draw plates. The cylinder is not completely closed. A longitudinal slit for the vapours to escape is retained. Both ends of the cylinder are plugged by a 20 mil wire spot welded into position. The free ends of the plugs are welded to stainless steel legs. The power supply is a 6-volt battery. Preliminary experiments with the oven empty under vacuum yielded temperature and current characteristics which are shown in graphs. A specimen of 347 stainless steel became red at 800°C and at 1,166°C it appeared to be hotter than the heater element which is attributed to exceptionally good black body conditions. At 1,530°C the steel flowed to one side of the oven. In similar experiments with chromic oxide the material reached a temperature over 2,700°C. The specimens used, though small, lasted a surprisingly long time. 1½ mg. of uranium tetrafluoride (UF_4) was studied at 800°C for 30 hours without the sample being exhausted. Two limitations to the usefulness of this type of oven were observed: In the case of tantalum the gettering power of this material interfered with the accuracy of the measurements during the study of the dissociation products of fluorides and oxides. In the case of platinum, peaks of that material could be observed in the mass spectrometer when the temperatures approached the melting point of platinum. Ovens of this type have been used successfully to produce thermal ions from lithium, strontium, caesium, barium and other materials.

Sommaire : Description d'un four pouvant être relié à la source d'ion du spectromètre de masse, et déstiné à vaporiser certains des spécimens métalliques et nombreux des oxydes difficilement évaporables.

Report by J. R. Walton *A.E.C. Report* No. ORNL-1473 January 1953

A Study on Diffusion Pump Oil by the Mass Spectrometer Analysis of the Final Vacuum 102/II

See Abstract No. : 90/II

Preparation of Specimens for the Electron Diffraction Camera 103/II

United Kingdom. The preparation of every material for the electron diffraction camera is a special case, the methods employed depending upon (1) the material and (2) the object of the investigation. It is not possible therefore to give a comprehensive paper covering all possibilities and the method of the article is to illustrate by examples the types of technique used and the precautions which must be observed. These, together with the literature references, are designed to enable the reader to devise suitable methods for any specific case. The subjects are dealt with under the following headings :—*Transmission* : Making Collodion Films; Powders, Suspensions and Deposits from Solution; Rubber, Oils, Greases; Metal Films; Oxide Films and Vitreous Substances. *Reflection* : Powders, Smokes, Crystals, Condensed Vapours; Oils, Greases; Compact Solids, Especially Single Crystals; Polycrystalline Solids. There are also sections on reactions carried out in the camera, and on precautions.

(*Author*)

Sommaire : Revue de différentes méthodes connues de préparation de spécimen pour le diffractographe électronique.

Article by P. R. Rowland *Vacuum* **3,** April 1953 136-150

Techniques for the Electron Microscopy of Crystals 104/II

United Kingdom. At the time when the electron microscope was introduced as an additional research tool it was essentially used for materials lacking in crystallinity and thus unsuitable for study by X-ray diffraction methods. A.o. biological materials belong to that group. X-ray diffraction methods yield information on the packing conditions in the crystal as a whole in the form of a statistical mean, but the precise arrangement of molecules at faces, edges and around lattice faults cannot be determined by it. Non-periodic structures of this kind in crystals can be examined successfully in the electron microscope and give important information on growth phenomena. Growth studies can be carried out in the light microscope by the Tolansky method but this method has only in one dimension a resolving power of 30 Å which is the appropriate resolving power of the electron microscope in all three dimensions. Both microscopical methods (light and electron) permit the investigation of crystal growth and thus represent complementary tools to the X-ray diffraction method

Vol. 3 1953

II — Vacuum Apparatus and Auxiliaries — II

Contd.

Abstract No. and References

which facilitates the determination of the basic molecular structure. The crystal surface of specimens prepared for the electron microscope must be molecularly clean. This is often difficult to achieve due to contamination by amorphous material. Proteins, for instance, crystallise very slowly so that the use of even a minute amount of solvent will leave an amorphous protein layer on the crystal after evaporation. If the proteins are water soluble a freeze drying technique can be used to overcome the difficulty. Details of the technique are given in its application to the study of β-lactoglobulin. A drop of the protein solution containing suitable micro-crystals is spread on a glass slide. When the micro-crystals have settled the slide is placed on a metal block, cooled by liquid air. Freezing takes place from the bottom upwards driving the protein solution on a metal block, cooled by liquid air. Freezing takes place from the bottom upwards driving the protein solution upwards until it reaches the top of the preparation. After careful vacuum drying the amorphous layer can be peeled off. Replicas must be used, if the crystals are too thick or too unstable for direct examination in the microscope. Wyckoff's pseudo-replica technique developed for use with proteins and water-soluble system materials is explained in detail. A layer of the specimen material is deposited on a glass slide, dried and shadow cast. Then very diluted collodion or Formvar is applied to the metal layer and the glass slide with its charge is immersed in water. The collodion or Formvar film with the shadow cast layer adhering to it, floats off and can be collected on screens, ready for use in the microscope after drying. Other methods described are, the silicon monoxide replica technique and a modification of Koenig's carbon replica technique. It has been claimed that specimens of a material with a melting point below 200°C would melt when exposed to the electron beam in the microscope. However, this happens only, if the material is thick enough to absorb a large percentage of the energy of the beam. The greater danger is the melting of the material during shadow casting. This can be counteracted by arranging for slow evaporation of the metal and by preventing excessive heat radiation from the crucible. Another effect frequently observed is that of pronounced granulation of the shadow cast layer. This is not only due to the heat produced by the electron beam. It can be promoted by traces of oil originating from the diffusion pump and condensing on the specimen. For chemical reasons the metal used in shadow casting should have minimum mobility on the condensing surface. Recently it was found that a 50/50 palladium nickel alloy gives much improved results on hydrocarbon specimens.

Sommaire : Description de méthodes modernes variées pour la réplique de monocristaux pour examen au microscope électronique.

Article by I. M. Dawson *Brit. J. Appl. Phys.* **4**, June 1953 177-181

Aluminium Oxide Films in Electron Microscopy : The Production of Replicas with a Great Resolving Power

105/II

France. Oxide films produced electrolytically on an aluminium surface have a uniform thickness of a few tens of Ångstroems, are transparent to electrons of an energy ranging from 50 to 100 kV and reproduce faithfully the conditions of the aluminium surface. If the surface is polished the oxide film shows no structure whatsoever but if the aluminium surface is corroded the oxide film will show the corresponding pattern of structure. Oxide replicas give a better resolution than any other type of replicas but can only be obtained from aluminium and light-alloy materials. The present article reports on a method of reproducing aluminium oxide films which is similar but in some respects simpler than the methods described by Hass and MacFarland. As a first step aluminium is deposited on the surface of the specimen to be examined to a thickness of more than 1 μ. Subsequently a few drops of a 1% collodion solution are spread over the deposit and the solvent is evaporated. Then the aluminium film and the collodion backing is detached from the specimen surface with the help of cellotape. The film assembly on the tape is connected to an electric supply of 14-18 V and submerged in a conventional electrolyte. The oxide film forms in a few seconds. After oxidation the film assembly is washed in distilled water and dipped in acetone in order to dissolve the collodion. Following a second washing process the film assembly is cut into small pieces to fit the electron microscope specimen grid and the pieces are dropped into a saturated mercury chloride solution to dissolve the aluminium. After drying the oxide film is ready for use in the electron microscope. As it is somewhat difficult to ascertain which is the upper side of the replica it is recommended to pre-shadow the oxide film before the aluminium base is dissolved. In order to obtain oxide films with no visible structure the aluminium film should be deposited on a clean glass surface. A number of micrographs are reproduced to show the effectiveness of the method. For instance, a surface etched chemically is compared with a surface etched cathodically. The former shows the typical corrosion picture whereas the latter demonstrates that cathodic etching removes the metal uniformly.

Sommaire : Méthode de preparation de répliques d'oxydes et examples de leur application.

Paper by C. Fert & R. Dargent *C. R. Acad. Sci., Paris* **235**, 22.12.52 1623-1625

A New Method of Making Electron Microscope Specimen Support Films

106/II

United Kingdom. It has been found that dipping an electron microscope specimen grid into a 1% solution of Formvar in chloroform, and allowing it to dry in air produces a very thin supporting film which is stronger than those wet-stripped from glass. On withdrawal from the solution the film is dried in a horizontal position. The thickness of the film obtained can be varied by changing the concentration of the solution from 0.5–1.0% and by varying the number of squares covered with the solution on withdrawal of the grid from the bath. By means of a photograph taken in an interferometer microscope it is shown that the film is uniformly thin in the centre of the mesh square over 70–80% of the total area. Near the edges it is thicker. As the film lies between the top and the bottom of the grid bars shadowing must be carried out at angles of $\tan^{-1}\frac{1}{2}$ and $\tan^{-1}\frac{1}{3}$ in order to cover the whole area of the film.

Sommaire. Brève description d'une nouvelle méthode de préparation de spécimen supports pour le microscope électronique.

Letter by D. E. Bradley *Nature* **171**, 13.6.53 1076-1077

The Breaking of Tobacco Mosaic Virus Using a New Freeze Drying Method

107/II

See Abstract No. : 52/IV

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

26 — MATERIALS — 26

Apparatus for Vacuum Firing of Porcelain in Dental Articles 108/II

See Abstract No. : 186/I

Arc-Melting of Non-Metallic Materials and a Re-Determination of the Beryllia-Thoria System 109/II

United Kingdom. An argon atmosphere arc-furnace, employing a non-consumable tungsten electrode and a water cooled copper hearth, is used to heat and melt a wide range of refractory systems. The furnace operates under an argon pressure of about 20 cm. Hg. The argon is first purified by passing over magnesium turnings at 600°C and further by arc-melting a piece of zirconium. The latter is pushed to the hearth rim by external manipulation of the electrode and replaced by a specimen or series of specimens. Power for the arc is drawn from a d.c. welding generator. With the specimen to be melted located in the centre of the hearth an arc is struck by lightly touching the electrode onto the hearth. To prevent the puncturing of the hearth by the initial power surge a striking power setting of 1 kW is used. When the arc is stabilised the electrode is moved to enable the arc to envelope the specimen which is thereby heated until at about two-thirds of its melting point it commences to conduct the arc current. Convection stirring ensures homogeneous mixing of different materials. Good contact between the refractory and the copper hearth minimises copper pick-up by secondary arcing. Of the several materials melted the following showed no change in their structure as determined by X-ray diffraction :—Al_2O_3, BeO, Cr_2O_3, ThO_2, V_2O_5, CaF_2, NaF and CaC_2. Both TiO_2 and Ta_2O_5 possessed good electrical conductivity after melting which was destroyed by heating the specimens in air at 1,000°C. MgO could not be melted due to excessivee vaporation. Melted CaO was only slightly attacked by 25% HCL during a two-hour immersion, the attack was appreciable after 24 hours. Iron oxide, C9 glass, porcelain and SiO_2 were all melted though the latter was difficult to melt owing to poor electrical conductivity in the molten state and rapid vaporisation. A re-examination of the BeO/ThO_2 system was made by melting point determinations on small beads. The beads were held partially molten in the arc and the boundary temperature between liquid and solid measured with a disappearing-filament pyrometer. A correction curve was obtained with refractory metals of known melting points. The temperatures obtained do not represent the true melting points but the form of the liquidous curve was obtained. A eutectic was located between 10 and 15 wt% BeO. X-ray diffraction photographs showed no intermediate compounds. The ThO_2 pattern was predominant in all photographs with the BeO pattern detectable in mixtures with 50% BeO and more.

Sommaire : Four à arc atmosphère d'argon utilisé pour la fusion de matières refractaires inorganiques.

Article by G. A. Geach and M. E. Harper *Metallurgia* **42**, May 1953 269-271

Improvements i.o.r.t. the Concentration by Evaporation of Heat Sensitive Liquors 110/II

See Abstract No. : 119/III

Carbon Dioxide 111/II

United States. Two dry salts react directly with each other in high vacuum to produce one thermally stable product and one thermally unstable product; the latter is decomposed to yield pure gaseous CO_2. Thus 53 g. pure, dry, anhyd. $PbCl_2$ was mixed with 9.67 g. pure dry, powd. $Ba–CO_3$, and the mixture was placed in a sealed container which was then evacuated to 10^{-5} mm. Hg pressure. The evacuated chamber was heated, and at 240–50° the pressure increased at the rate of about 1 mm. Hg per min. At slightly higher temperatures the rate of pressure increase was more rapid. The CO_2 is formed when the reaction product, $PbCO_3$, decomposed.

(*Chemical Abstracts*)

Sommaire : Méthode de production de CO_2 gazeux.

W. W. Miller, J. Turkevich, N. Zwiebel & A. E. C. *U.S. Pat.* 2,622,008

Paper for Punched Cards 112/II

See Abstract No. : 147/I

Helium Diffusion Through Glass 113/II

See Abstract No. : 43/IV

The Limiting Negative Pressure of Mercury in Pyrex Glass 114/II

See Abstract No. : 93/II

Mercury Vapour as a Health Hazard 115/II

United Kingdom. Mercury, although a heavy metal, is quite volatile. At 65°F it has a vapour pressure of 0.001 representing a saturation value of 10,400 μg.p.m^3 of air. Mercury vapours are poisonous. The symptoms

Vol. 3 1953

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

produced by chronic inhalation of mercury vapours are well known and will only clean up slowly if the patient is removed from contact with the metal. Atmospheric concentrations as high as 1,340μg or even 2,050μg/cm^3 air have been recorded in certain instances in badly-ventilated workshops where mercury was handled. 100/μg/cm^3 air is the maximum considered to be safe. Mercury vapours are colourless and odourless. The only methods known (Nordlander or Buckell) for the determination of their concentration in the atmosphere of a room require the services of an expert. A mercury vapour detector has now been constructed which can be operated by an unskilled person. The apparatus consists of a cylinder in a horizontal position fitted with a quartz mercury discharge tube at one end and a photocell at the other. The air to be tested is drawn along the tube by a fan. Normally, the cell registers the full intensity of the radiation received from the discharge tube but if the air contains mercury vapour the radiation intensity received by the photocell is reduced. This effect is based on the well-known phenomenon that the resonance radiation emitted by a substance is absorbed by the vapour of the same substance. The micro-amperemeter of the instrument is directly calibrated to given concentrations of mercury vapour. There is a fine and a coarse scale of sensitivity covering the range from 2μg. to 1,000 μg./m^3. The instrument weighs 27 lb. and consumes 75 W.

Sommaire : Un détecteur de vapeur de mercure développé récemment est décrit.

Article by D. A. Jones *The Times* (*Industr. Suppl.*) 7, May 1953 33

Direct Determination of Oxygen in Rubber — 116/II

O See Abstract No. : 176/I

28 — APPARATUS COMPONENTS — 28

A Vacuum Valve to Provide Small Controlled Leak Rates — 117/II

See Abstract No. : 137/I

Glass Leak and Control Valves — 118/II

See Abstract No. : 136/I

A Large Unwelded Vacuum Gate Valve — 119/II

Japan. A six-inch straight-through valve is described which is based on a design originally reported by L. D. P. King (see *Rev. Sci. Instrum.*, 19, 1948, 83). In order to avoid welding and soldering of the precision-made metal plates which might cause distortion, rubber gaskets have been used throughout for sealing purposes. The side plates of the valve casing—a box oblong in shape—are fixed to each other by a large number of small screws. The vacuum pipes are joined to the two opposite broad faces of the casing. The gate plates have a plane surface on the seating side and are tapered on the inside. The two plates are separated by a wedge moving on a spindle operated from outside. In the closed position the wedge, in contact with the tapered surfaces of the gate plates, presses the latter against the side plates of the valve casing. In order to open the valve the spindle is turned in a direction which causes the wedge to release the pressure on the gate plates but the gate plates remain stationary until the wedge travelling along the spindle hits a pin fitted to the plates. Then, if O rotation of the spindle is continued, the gate plates are withdrawn from the openings in the valve casing.

Sommaire : Discussion de la construction d'une vanne à vide à passage directe.

Article by S. Asao & K. Muramatsu *J. Sci. Instrum.* 30, June 1953 209-210

A Large-Diameter Reciprocating-Action Vacuum Valve — 120/II

United States. A double-piston valve has been designed for laboratory vacuum systems eliminating the need for the use of glass stopcocks, in the lines connecting the system to the pump. The valve consists of a 1-inch diameter Pyrex tube, 19½ inch. long, sealed at one end. Two aluminium pistons are inserted in the tube, fixed to a common stainless steel rod which extends through an air-tight gland in a cap sealed to the open end of the tube. 'O'-rings are fitted to the circumference of the pistons which are compressed by about 0.020 inch. Connections to the system to be evacuated, the fore pump and the diffusion pump are sealed to the Pyrex tube in such a manner that three operating positions of the pistons are obtained and the forces on the pistons are continually balanced during all operations :—(*a*) the rod is pulled out. The vacuum system is connected to the fore pump. (*b*) The rod is pushed in. The system is connected to the diffusion pump. (*c*) Intermediate position of the rod. The system is connected to atmosphere but the pumps remain protected. The overall movement of the pistons is 5½ inch. The valve works satisfactorily at pressures of about 10^{-7} mm. Hg. Reference is made to a publication by Shatford (see Vol. 2, Abstract No. : 152/II).

Sommaire : La construction d'une vanne permettant de raccorder l'enceinte à pompe soit au vide poussé ou au vide moyen, sans robinets, est décrite.

Report by G. L. Fox *A.E.C. Rep. No.* MLM 850 May 1953

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

A Design of Pressure/Vacuum Valve

121/II

United Kingdom. In a closed circuit containing liquid metal it is necessary to blanket the free surfaces of the metal with an inert gas to prevent oxidation of the metal. It must also be possible to evacuate the system before the blanket gas is admitted. The use of conventional valve types employed in gas lines was unsatisfactory because of leakage across the valve seating. The valve described in this report uses sliding ' O '-rings to effect all seals. It is of the right-angle type and consists essentially of the valve body made of stainless steel, a piston made from mild steel nickel-plated and a cap made from duralumin engaging in the thread machined on the outside of the valve body. The piston carries two ' O '-rings resting in grooves. In the closed position one ' O '-ring seals off the valve port the other acting as a gland. To open the valve the piston is moved into the upper portion of the body by turning the cap. In order to avoid damage to the ' O '-ring as it passes the port in the body, the diameter of the piston bore is increased in that area. The maximum pressure against which the valve can be closed is 3–35 lb./sq. inch. Reference is made to a publication by Shatford (see Vol. 2, Abstract No. : 152/II).

Report by F. L. Speed *A.E.R.E. Rep.* No. ED/R 1302 June 1953

Sommaire : Détails d'une vanne pression/vide pour utilisation dans un système clos en présence de métal en fusion.

29 — MISCELLANEOUS APPARATUS — 29

Design and Use of an Electronic Pressure Controller

122/II

United States. The determination of vapour-liquid equilibria requires a very delicate control of pressure. The pressure control described in this article has been specially designed for use in connection with equilibrium stills of laboratory size and holds the pressure constant indefinitely at any value of subatmospheric pressure or pressures slightly above the atmosphere. It consists essentially of a U-tube manometer one side of which is connected to a reference pressure bulb and the other to the system to be controlled. The manometer liquid, an electrolyte, acts as a seal to the gas in the reference bulb and by submerging the whole apparatus in a small constant-temperature bath the volume of the reference gas is maintained constant for any length of time. The apparatus is fitted with three electrodes a common electrode in permanent contact with the liquid and an electrode A and an electrode B at the top of the two arms of the ' U '. If the fluid makes contact with electrode A, the pressure in the system is excessive and if it makes contact with the electrode B the pressure in the system is below the required value. As a result an electrical circuit is closed and a signal transmitted to the main controller. A complete circuit diagram of the latter is shown and details of its operation are given. On receipt of the signal the main controller opens, *via* a relay, one of two tyre-valves situated in a manifold kept at reduced pressure and in a manifold containing compressed air respectively. The valve remains open until the system has been restored to the control point. A separate control circuit is used to hold the water bath temperature constant. A mercury thermostat acts as the sensing element. The heat source is a light bulb. In the particular arrangement the temperature can be controlled with an accuracy of ± 0.02°C.

Article by F. J. Digney & S. Yerazunis *Analyt. Chem.* **25**, June 1953 921-923

Sommaire : Appareil de réglage automatique de la pression qui a été développé pour utilisation dans la détermination de l'équilibre vapeur-liquide qui nécessite une réglage très fin de la pression.

Increased Sensitivity of Leak Detection with Hydrogen

123/II

United States. One standard method of leak detection consists of admitting hydrogen to the area suspected to contain a leak and recording the change in the reading of an ionisation gauge attached to the system, when the hydrogen penetrates into the interior. Although the ionisation gauge is less sensitive to hydrogen than air, hydrogen is a gas suitable for the purpose because it enters a small leak at a greater speed than air and is usually pumped out at a rate sufficiently different to produce a conspicuous net increase in gauge reading. The change in gauge reading can be made greater still by reducing the speed of the pump, *i.e.*, by lowering the voltage of the heater supply. A graph is given showing the variation of the readings recorded by the gauge as a function of the heater voltage for the three cases :—(*a*) Vacuum at leak, (*b*) air at leak and (*c*) hydrogen at leak. The variations noted can be taken as an indicator of the sensitivity of the gauge for leak detection and can be expressed therefore by the ratio :—

$$\frac{(\text{reading-hydrogen at leak})-(\text{reading-air at leak})}{(\text{reading-air at leak})\quad -(\text{reading-vacuum at leak})}$$

A second graph gives the sensitivity of the gauge for leak detection in terms of that ratio plotted against heater voltage, showing an optimum between 90 and 100 V. It has been noted that the pressure is not always stable at low heater voltages and for that reason it may be necessary to determine the optimum voltage individually in each particular system.

Note by G. A. Alers, J. A. Jacobs & P. R. Malmberg *Rev. Sci. Instrum.* **24**, May 1953 399-400

Sommaire : Méthode d'augmentation de la sensibilité de la recherche de fuite, utilisant l'hydrogène comme gaz révélateur.

Calculation of the Rate of Diffusion of Hydrogen through a Plate of Palladium

124/II

See Abstract No. : 44/IV

II — Vacuum Apparatus and Auxiliaries — II
Contd.

Abstract No. and References

A Miniature Helium Liquefier-Cryostat of Cascade Type

125/II

United Kingdom. A miniature helium liquefier and cryostat in one unit has been developed to be made available to the experimenter as an individual source of supply of liquid helium, when and where he requires it. The cascade principle has been chosen for the liquefier design and the Joule-Kelvin method is used at both the liquid hydrogen and the liquid helium stage. As a result the starting time for liquefaction is reduced and further liquid helium quantities can be obtained at any time without warming to hydrogen temperature. A small oil-free gas tight compressor operating in a closed helium circuit is employed to counteract possible contamination. The liquefier is constructed of metal and provides four stages of cooling. The main vacuum chamber is submerged completely in liquid oxygen held in an open Dewar vessel. The chamber contains three smaller vessels. The first is maintained at 62°K by liquid oxygen boiling in it at 1 cm. pressure. The hydrogen pre-cooled in this vessel expands into the second vessel maintained at 20 cm. Hg pressure. The high pressure helium pre-cooled in the second vessel expands into the third vessel. This vessel is protected by a radiation shield. No liquid air is used for pre-cooling. Details of the construction are given in an appendix to the article. The plant has the following special features:—A valve, details of which are given, keeps the level of boiling oxygen in the first inner vessel constant by regulating the supply of liquid oxygen from the Dewar vessel. Initially the second and the third inner vessels are cooled by circulating helium through its own high pressure line, through the expansion valve and through a special line by-passing the return exchanger. Only when the temperature of 62°K has been obtained is it necessary to start the circulation of the hydrogen. Once started, the operation of the hydrogen circuit is fully automatic. If the flow of helium stops, the second inner vessel cools down to below 16.5°K and as soon as it is full the incoming hydrogen solidifies at the expansion valve which interrupts the flow. But if the vessel is empty or the passage of helium resumed then the valve unfreezes and allows more hydrogen to enter the vessel. This arrangement prevents the excessive production of liquid hydrogen. The operation of the plant has been timed, details of which are given. Liquid helium was obtained 32 minutes after the start. Subsequently helium is liquefied at a rate of 2.5 cm^3 per min. This corresponds to 85% of the theoretical yield. If the operation of the plant is continued for 10 minutes after the first liquid helium has become available, a quantity of liquid helium is produced enough to cover 5 to 10 hours working. Hydrogen is consumed at a rate of 35 litres for every 10 cm^3 of liquid helium produced. Another 40–50 l./hr. are required to compensate for evaporation losses. Assuming a 10-hour working day one standard cylinder of hydrogen will last two days. The consumption of liquid oxygen is about 8 l./day. The total quantity of helium in the circuit is about 200 l.

Sommaire : Un cryostat liquéfacteur d'hélium miniature a été développé en vue de rendre de l'helium liquide disponible pour l'expérimentateur quand il en a besoin, et le rend indépendant d'une source de ravitaillement centrale.

Article by P. F. Chester & G. O. Jones *Proc. Phys. Soc.* **66**, April 1953 296-300

Improvements i.o.r.t. Vacuum Insulated Systems for Conveying or Storing Liquefied Gases

126/II

United Kingdom. The storage of liquefied gases is normally effected in vessels which consist of an inner container holding the liquefied gas and an outer shell, the space between the two walls being evacuated. If this space is filled with a suitable powder, the same degree of thermal insulation can be obtained with a lower vacuum but the presence of the powder affects adversely the rate, at which evacuation can proceed. To overcome this difficulty it is proposed to surround the inner container with a layer of powder either by bonding it to the outer wall of the container or by providing a sheath of fabric, leaving a substantial part of the space between container and shell unobstructed in order to facilitate proper evacuation. The new design is particularly recommended for application to insulated pipe sections.

Sommaire : En vue de vaincre certaines difficultés dans la production de tuyeaux isolés à la poudre, servant au transport de gaz liquéfiés, il est suggéré certaines améliorations dans la construction.

R. S. B. Madeley, N. W. Roberts & British Oxygen Co. *Brit. Pat* 690,748

Mercury Vapour as a Health Hazard

127/II

See Abstract No. : 115/II

Potash Refining in France

128/II

See Abstract No. : 133/III

III — Vacuum Processing Techniques — III

Abstract No. and References

30 — EVAPORATION — 30

Research into Metals 97/III

See Abstract No. : 180/I

Metal Evaporation in High Vacuum 98/III

Germany. The preparation of thin films by vacuum evaporation is gradually taking the place of all other known methods because it is a comparatively simple and easily controllable process. Coating chambers are now available in sizes ranging from that of a normal glass tube to several metres diameter. The operational pressure must ensure a mean free path greater than the distance between source and support, *i.e.* must be of the order of 10^{-4} mm. Hg. But even at this pressure adsorption layers are still formed on the support which contaminate the metal deposited. For that reason dry ice traps should be used, if oil diffusion pumps are employed and liquid air traps if mercury pumps are employed, in particular where gold or other metals tending to amalgamate are evaporated. A theoretical treatment of the evaporation from a point source is given. The speed at which the thickness of the deposit grows depends on the distance between source and support, the evaporation rate and the temperature and surface condition of the substrate. A table is shown originally published by Dushman giving the evaporation temperatures, melting points and masses evaporated from 1 sq. cm.p.sec. for 53 metals. Added to this is information on suitable evaporation sources. Where the evaporation temperature of the metal is below its melting point the metal can be directly evaporated from wire consisting of that metal. In all other cases filaments or boats of materials of appreciably higher evaporation temperature are used which are either resistance-heated or high-frequency heated. A third form of heating the material is electron bombardment. During the first 5–10 minutes of operation, while impurities are volatilised, the support should be shielded from the source. The standard materials for filament sources are tungsten, molybdenum or tantalum. If the material to be evacuated wets the filament material it is placed in the form of small rods into the filament. These rods may short-circuit parts of the filament resulting in overheating. This can be overcome by plating the material to be evaporated onto the filament. Materials such as silver and copper do not wet the standard filament materials mentioned, but filaments can still be used as long as they are wound sufficiently closely to prevent the metal penetrating the gaps; alternatively boats should be used. Alundum crucibles with built-in tungsten heater elements (melting point 2,000°C) are often used. Methods of fabricating such crucibles are described. Graphite and molybdenum crucibles can be heated electronically to 3,500°C using a tungsten filament as the cathode. A theoretical study of the geometrical distribution of the deposit on the substrate follows. Various source arrangements to produce a uniform coating on the support are discussed. A special method of thickness control proposed by Jacquinot is mentioned who mounted the support on a small truck which was moved electromagnetically backwards and forwards across the molecular beam. With the help of suitable diaphragms placed in front of the support, gray wedges for photometric purposes could be produced by this method. The following section of the article is devoted to film formation and film structure. It appears that surface migration is a function of the melting point of the metals. Therefore in many cases surface migration of the more volatile metals can be reduced by depositing a (approx.) 10^{-2} molecular layer of a higher melting point metal first. Thorough cleaning of the support is necessary. A glass surface is considered clean if, breathing on to it, a uniform black skin appears as distinct from the normal breath condensate. Chemical means of cleaning are washing with soap, followed by washing in distilled water and finally dipping in aqua regia. As some glasses are liable to be attacked by chemicals, polishing with $CaCO_3$ or alcohol is preferable. Rubbing with cottonwool will invariably add new fatty layers to the glass unless the cottonwool has been kept in benzene. The most satisfactory method is ion bombardment after thorough chemical cleaning. Where the glass plates are small, it is sufficient to pass them through a Bunsen flame. But larger plates should be placed in the vacuum chamber and exposed to a glow discharge of about 3–5,000 volts and 15–100 mA. in an atmosphere of oxygen or hydrogen at a pressure of 10^{-3} or 10^{-4} mm. Hg for about 10 minutes. It is advisable to to coat the plates while they are still warm in order to prevent the formation of fresh adsorption layers. A suitable arrangement for the purpose is shown in a photograph. It should be noted that ion or electron bombardment may produce a rough surface on the support. This is undesirable for certain types of research work. The article concludes with a short survey on protective films and unsupported films.

Sommaire : Revue generalé des connaissances actuelles sur l'évaporation de films métalliques pour des besoins scientifiques.

Article by S. Methfessel *Glas und Hochvakuum-Tech.* **2,** April 1953 167-175

Resistance Heaters for Vacuum Evaporation 99/III

United Kingdom. The design of heaters for thermal evaporation of substances in vacuum is considered. Three designs of heaters and useful adjuncts are described, and means for mounting them are discussed.

(*Nuclear Science Abstracts*)

Sommaire : Description de trois constructions de sources d'évaporation.

Report by J. G. S. Biram *A.E.R.E. Rep.* No. EL/R1065 December 1952

Evaporation of Germanium Films from a Carbon Crucible 100/III

United States. The rate of evaporation of germanium in the temperature range from 1,100 to 1,550°C was determined. The pressure during evaporation was 0.1 μHg. The substrate was a sheet of mica. A resistance-heated carbon boat was used as crucible. The boat was specially designed to keep the charge at a high and

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

constant temperature. An illustration of the assembly is shown. The charge was 50 mg. per run. The distance between the germanium surface and the crucible and the substrate was 37.5 mm. The evaporation rate was determined by measuring the transmission through the mica sheet by means of a germanium photocell and plotting the measured values against time. The curves obtained in this manner show a series of maximima and minima. The rate of deposition (p_o) can then be calculated from : $p_o = \lambda/2\eta\tau$. In the particular case the value used for λ is 1.7 micron and η, the respective refraction index, is 4.6. τ is the period between two successive maxima. At temperatures of 1,450°C and higher τ is so small that the optical method becomes impractical and the deposit had to be weighed in order to determine the evaporation rate (p_m). p_m represents the average across the whole mica sheet whereas p_o is applicable to the centre of the sheet only. To line up the results of the two methods a number of determinations were made by both methods at low temperatures which yielded the calibration factor required to adjust the p_m values accordingly. The results are given in a table which is partly reproduced below.

Rate of Evaporation

Run number	*Temperature °C*	*Weight of Film* Deposited (mg)*	*pm from Mass Data (g/sec.)*	*po from Optical Data (Å/sec.)*	*Period τ (sec.)*	*Ratio Po/pm (cm/g)*
4	1,114	3.1	0.252×10^{-6}	0.27	6,750	1.1×10^{-3}
12	1,259	5.6	3.46	3.6	510	1.0
2	1,332	11.1	12.3	13	146	1.1
6	1,477	10.5	175.0	180		
9	1,550	9.2	307	310		

* The deposited film decreases in thickness from centre to outside. In order to get the film thickness in the centre multiply value in this column by calibration factor 0.01 cm./g.

Fluctuations of the pressure at the pressure level considered do not appear to affect the results. According to the data obtained from the experiment the activation energy of evaporation is 3.5 ev. This value agrees with the determinations by Searcy.

Sommaire : La vitesse d'évaporation de germanium depuis des creusets de carbone a été determinee dans le domaine de 1,100 à 1,500°C.

Article by K. Lehovec, J. Rosen, A. MacDonald & J. Broder *J. Appl. Phys.* **24**, May 1953 513-514

A Note on ' Metallic Shadow Casting Using a Nozzle System ' — 101/III

Japan. The author evaporated pure titanium onto glass and found by means of electron diffraction that the deposit contained tungsten originating from the evaporation source. In an article by Hibi (see Vol. II, Abstract No. 181/III) a nozzle device for shadow casting is described. An illustration in this article shows a specimen shadow-cast with titanium. The deposit does not appear to be characteristic of titanium. The author points out that in certain circumstances the vapour conveyed through such a nozzle might consist predominantly of tungsten and this condition must be taken into account in the assessment of the shadowing qualities of the various materials.

Sommaire : Critique abrégée de certaines déclarations dans la publication mentionée au No. 181/III concernant l'ombrage à l'aide d'une tuyère.

Letter by S. Yamaguchi *J. Appl. Phys.* **24**, June 1953 815

Study of the Crystalline Grain in Thin Films of Gold or Palladium or Gold-Palladium Alloys Prepared by the Thermal Evaporation Techniques — 102/III

See Abstract No. : 161/I

On the Structure Loosening of Metal Films Deposited from the Vapour — 103/III

Japan. High-purity bismuth films deposited at 10^{-4} mm. Hg pressure on copper-supported thin glass slides showed structure loosening at temperatures appreciably lower than that of the melting point of the bulk material, when examined in the electron diffraction camera. All precautions to avoid excessive heating of the specimen by the electron beam were taken. A graph showing the temperature of structure loosening plotted against film thickness shows that structure loosening occurs at temperatures near the melting point (=272°) only if the thickness exceeds 3μ. Examining the films in the microscope co-aggregation could be observed, where films less than 0.5μ thick were heated at the structure loosening temperature and then cooled, but no such change could be observed in thicker films. The grain size of the crystals in the surface of thicker film was approximately 1μ and not affected by a change of energy of the beam in the diffraction camera. The grain size, therefore, cannot have any influence on the temperature at which structure loosening occurs. Nor does it appear that the orientation of the crystals in the deposit is related to the value of the temperature of structure loosening. Finally, neither the evaporation rate nor the temperature of the substrate during evaporation

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

seemed to have any appreciable influence on the temperature of structure loosening. Experiments with tin deposits produced similar results. The authors conclude that the value of the temperature, at which structure loosening occurs, is essentially controlled by the thickness of the film.

Sommaire : Durant un examen au diffractographe électronique l'on a observé un relâchement de la structure du film de bismuth, celà à une temperature nettement inférieure à celle du point de fusion de métal en masse. Les causes du phenomène ont été étudiées.

Note by Y. Fujiki & R. Suganuma *J. Phys. Soc. Japan* **8**, May-June 1953 427-428

Aluminium Films in Electron Microscopy: The Producation of Replicas with Great Resolving Power 104/III

See Abstract No. : 105/II

Positive and Negative Ions in Cathode-Ray Tubes 105/III

See Abstract No. : 153/I

The Preparation of Titanium Dioxide Beam-Splitters of Large Surface Area 106/III

United Kingdom. Titanium dioxide beam-splitters have been prepared, for use in a three-colour television monitor, by heat oxidising pure titanium films in air. Their advantages as compared with the more ideal dichroic beam-splitters are discussed. A short description of the high vacuum apparatus used for producing titanium films of uniform thickness is given. A method of overcoming the flatness distortion encountered in the heat oxidation of titanium films on glasses of large surface area is reported.

(*Authors*)

Sommaire : Des filtres semitransparents au dioxide de titanium ont été préparés par oxidation de films purs de titanium à chaud à l'air libre. Leurs avantages sont comparés à ceux des filtres semi-transparents dichroiques.

Letter by L. Holland, K. Hacking & T. Putner *Vacuum* **3**, April 1953 159-161

On the PbS Photoconductive Cell 107/III

See Abstract No. : 163/I

Multi-Layer Dielectric Reflecting Films at Wavelengths Between 4,000 and 4,600 Å. 108/III

See Abstract No. : 164/I

On the Choice of Measurements to be Made for Simultaneous Determination of the Optical Constants and Thickness of Thin Absorbing Films 109/III

France. An approximation formerly proposed by H. Wolter relating reflectivities, absorptions and transmission for thin films is re-examined graphically. Using existing experimental data it is confirmed that the Wolter relation holds for thickness less than 0,027 of the wavelength of the light used. Further in the region of optical constants for Ag in the visible the relation holds whatever the thickness. The method described enables constants and thickness to be obtained with only three measurements.

(*Science Abstracts*)

Sommaire : Description d'une méthode facilitant la détermination des constantes optiques et de l'épaisseur de couches minces absorbantes à l'aide de trois mesures seulement.

Article by D. Male *C. R. Acad. Sci., Paris* **235**, 22.12.52 1630-1631

Improvement of Grating Efficiency in the Vacuum Ultra-Violet by Platinising 110/III

See Abstract No. : 167/I

The Optical Constants of Gold and Silver in the Ultra-Violet 111/III

See Abstract No. : 166/I

Adsorption by Evaporated Copper Films at 78°K. Part I. Krypton and Hydrogen 112/III

See Abstract No. : 40/IV

Adsorption by Evaporated Copper Films at 78°K. Part II. Krypton and Oxygen 113/III

See Abstract No. : 41/IV

Vol. 3 1953

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

High Vacuum Metallising of Metals and Plastics

114/III

United Kingdom. The present state of vacuum metallising is surveyed in all its aspects. Vacuum metallising has established itself as an industry because coating by evaporation is either the only method available of coating an article (*e.g.* coating of non-conductors), or the cheapest means of applying a coat of special decorative value (*e.g.* hammer or wrinkle finish). The basic sequence of operations is described and the working principle of the equipment required is discussed. The smallest coating plant mentioned is operated by a 9-inch. diffusion pump backed by a mechanical pump and the largest mentioned has a 48-inch. vacuum chamber, served by 2 oil diffusion pumps, 3 mechanical pumps and 1 booster pump. Production arrangements are discussed and the layout of a complete processing department is given. The uses of a riser rod as work-carrying device are explained (see Vol. II, Abstract No. 4/III). Two typical examples of production output are given based on the use of the large plant and 3,500 sq. ft. floor area being available. Assuming 3 coating cycles p. hour 11,000 small plastic buttons can be metallised p. hour, using 288 riser rods in all. Toy pistols can be coated at a rate of 936 p. hour. Running costs are given in terms of consumption :—Pumps of the 48-inch. plant—18.50 kW, ovens and dipping machines—14 kW, lacquer—2 gal p. hour, tungsten filaments—50 ft. p. hour, labour—4 operators (excl. progress). Finally, reference is made to continuous coating. In special plant rolls of cloth, paper, fibrous film or plastic material containing up to 1,500 ft. of material 20 inch. wide can be coated with aluminium at a rate of 20 ft. p. minute.

Article by Anon. *Industr. Finish.* **5**, March 1953 546-561

Sommaire : Revue généralisée de la métallisation sous vide de métaux et plastiques.

The Coating of Plastics by Vacuum Evaporation for Functional and Scientific Purposes

115/III

United Kingdom. Vacuum, metallising of plastics for decorative purposes has received wide publicity in recent years but less is known regarding scientific applications of the method. The present paper deals essentially with optical and electrical applications. The two main plastic materials used for optical compounds are :—acrylic and polystyrene. Lenses made from these materials may require an anti-reflection coating and mirrors may require a (vacuum evaporated) reflecting film. The method of ion bombardment can be used for cleaning the surfaces prior to coating but the intensity of the bombardment should be carefully controlled otherwise discolouration occurs due to decomposition of the material at the surface as a result of high-velocity ions hitting the surface. Another difficulty is presented by the lack of mar resistance of both materials. Polystyrene, the harder of the two, has a mar resistance still lower than glass. Various methods have been suggested to overcome this difficulty but in the experience of the authors none counteracts effectively the inherent softness of these surfaces. Distortion of the base has been noticed as a result of absorption of moisture from the atmosphere through the non-metallised face of the plastics. These conditions tend to limit the application of optical coatings to the interior surfaces of sealed components. Speaking of practical applications it is stated that where reflectors are to be run with high-wattage lamps the high heat insulation of plastics must be taken into account which will cause the temperature of the reflecting film to rise unduly and result in a deterioration of its reflectivity value. The normal method of suppressing unwanted surface reflectivity applied to glass components can be used for optical plastics materials as well, but single and anti-reflection coatings on plastics are rather ineffective as shown in a table reproduced below as there is no plastics with suitable properties

Effect of $\lambda/4$ MgF_2 Film on the Reflectivity of Perspex

λ(Å)	*Untreated Perspex*		*Bloomed Perspex*	
	Transmission %	*Reflection** %	*Transmission* %	*Reflection** %
4,600	92	8	96	3
5,300	92	8	97	2½
5,700	92	8	97	2½
6,000	92	8	96	3

* The reflectivity is that of the two boundary surfaces, both of which have been anti-reflection coated.

available which has the required low refractive index. However where a series of superimposed reflecting surfaces are used their application is still worth while. The electrical properties of thin metal films are different from those of the bulk metal. Some of their conducting properties are conditioned by the nature and behaviour of the substrate. For instance, plastics distort, if exposed to excessive heat radiated from the evaporation source resulting in crazing of the deposited film. Special crucible techniques are required in the case of chromium. Instead of loading the tungsten filament with chips of the metal, the tungsten should be electro-plated with the metal. This facilitates faster evaporation at a lower temperature. One important application is anti-static coatings. Reference is also made to Preston's method of sputtering metal oxide film of low absorption onto glass to act as resistance heaters but this method is not practical in the case of plastics due to the temperature rise during sputtering. As an alternative, iron oxide films and chromium films were deposited

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

on Perspex by the authors. Their relevant properties are shown in a table, reproduced below. Finally the of thin conducting films on plastics to serve as surface electrodes is mentioned.

Optical and Electrical Properties of Anti-Static Films

Chromium			*Iron Oxide*		
T	*A*	*Resistivity Ohms/cm.²*	*T*	*A*	*Resistivity Ohms/cm.²*
85	6%	1.2×10^{10}	87	3.2%	9.5×10^{9}
77	15%	1.5×10^{6}	84	7.2%	8×10^{9}
			80	10.5%	3.5×10^{9}
			70.5	13.0%	2×10^{5}

A is equal to 100–R–T, where R and T are the reflectivity and transmissivity respectively.

Article by L. Holland & N. Sutherland *Plastics Progr.* 1953 361-380

Sommaire : Revue de la métallisation de plastiques pour des buts fonctionnels, avec référence particulière aux application optiques et électriques.

31 — CATHODIC SPUTTERING — 31

Improvement of Grating Efficiency in the Vacuum Ultra-Violet by Platinising 116/III

See Abstract No. : 167/I

33 — IMPREGNATION — 33

Good Carpets from Good Cops 117/III

See Abstract No. : 146/I

36 — DRYING — 36

New Field for High Vacuums 118/III

See Abstract No. : 169/I

Improvements i.o.r.t. the Concentration by Evaporation of Heat Sensitive Liquors 119/III

United Kingdom. The concentration plant described is used for the processing of fruit juices, coffee extracts etc., the aromatic constituents of which are particularly heat-sensitive and, for that reason, have to be withdrawn at an early stage in the process. The construction of the plant is similar to that described in *Brit. Pat.* 498,305 and 575,118. But steam was used as a source of heat in the earlier design whereas a refrigerant such as ammonia or sulphur dioxide circulating in a closed gas-liquid circuit is employed in the present plant. The refrigerant leaving the compressor incorporated in the circuit supplies heat to the two evaporators of the plant. The partially liquefied refrigerant then reaches a heat-exchanger operated with cooling water. Subsequently it is fed to an expansion valve which vaporises part of the refrigerant and returns the vapours to the suction side of the compressor. The part of the refrigerant not liquefied in the expansion valve is circulated to the condensers of the plant where it takes up enough heat from the processed material to be vaporised before it reaches the suction side of the compressor. A flow sheet of the process is given. As a first step the liquor to be concentrated is fed to a pre-heater where its temperature is raised just above the boiling point of the liquor under the vacuum existing in the circuit. The liquor then passes into a flash separator where the aromatic constituents are separated from the liquor, passed to a condenser and extracted in liquid form. The liquor leaving the separator is fed to a climbing film evaporator where the main concentration process takes place. The vapours obtained at this stage are separated from the concentrated liquor, condensed and extracted through a separate pumping line. The system described has a lower overall power consumption and lower operating temperatures than the steam-operated plant.

Sommaire : Appareillage pour la concentration par évaporation de solutions sensibles à la chaleur qui utilise de l'ammoniaque ou des réfrigérants semblables comme échangeur de chaleur.

J. A. Reavell & Kestner Evaporator and Engineering Co. Ltd. *Brit. Pat.* 687,106

Vol. 3 1953

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

37 — METALLURGICAL PROCESSES — 37

Research into Metals 120/III

See Abstract No. : 180/I

Vacuum Furnaces for Melting or Processing of Metals 121/III

United States. Vacuum furnaces are now used for processing on an industrial scale and have the following principle advantages :—1. They facilitate refining of active metals such as titanium or zirconium. 2. In certain instances they are more economical than controlled atmosphere furnaces. Hydrogen, for instance, is expensive. 3. Vacuum-cast common metals show a reduced gas content. There are three types of vacuum furnaces : Resistance, induction and arc furnaces. The first group has normally a maximum operational temperature of 1,850°F. Special designs fitted with molybdenum heater elements reach temperatures up to 2,750°F. A horizontal type and a vertical type of resistance furnace are shown in sketches. Induction furnaces are chiefly used for the casting of metals with a melting point above 1,850°F and for degassing and annealing. A design providing a stationary crucible and another providing a tilting crucible are described. Reference is made to the melting furnace described in Abstract No. 39/III. Arc furnaces are used for melting metals with a very high melting point or active metals such as zirconium which would react with the crucible material. There are two principally different designs : (*a*) the arc operates between the metal and a permanent graphite, molybdenum or tungsten electrode, (*b*) a consumable electrode is formed by the metal itself which is to be melted. The latter method is preferable where highly reactive metals are treated.

Article by G. J. Crites *Metal Progr.* **63**, May 1953 161-166

Sommaire : Les caractéristiques les plus importantes et les applications de 3 types de fours à vide, c.à.d. à résistance, induction ou arc, sont discutées.

Electrical High Temperature Furnaces, Fitted with Molybdenum Resistance Heaters 122/III

See Abstract No. : 183/I

A High Temperature Resistance-Heated High Vacuum Furnace 123/III

United Kingdom. Most of the technical furnaces used for heating objects of reasonable size to high temperatures under very high vacuum are at present heated by high frequency induction, though the equipment is expensive and often difficult to obtain. An experimental 10 kW vacuum furnace with a resistance heating element is described in this report. It can be assembled from standard vacuum coating equipment to operate directly from the a.c. mains through a transformer. Promising operational results have been obtained and the design is suitable for the construction of larger furnaces on the same principle. The cost is about half that of an induction furnace of similar size and performance.

(*Authors*)

Article by W. J. Kramers & F. Dennard *Vacuum* **3**, April 1953 151-158

Sommaire : La construction d'un four à vide expérimental chauffé par une résistance de 10 kW est décrite et des indications générales sont données au sujet des avantages de l'usage du chauffage par resistance sur le chauffage par induction.

A Versatile Vacuum Melting Furnace 124/III

United States. A specially designed vacuum induction melting furnace operating at pressures 0.03 to 1.0 micron is described. The furnace capacity is 1/2 to 15 pounds of steel. The metal may be cast in vacuum by tilt pouring, bottom pouring, or centrifugal casting.

(*Author*)

Report by R. V. Gray *A.E.C. Rep. No.* KAPL-865 Jan. 1953

Sommaire: Four à vide a induction ayant une capacité entre 1/4 et 7.5 kg., pouvant être utilisé pour le moulage centrifuge ou alternativement pour coulée par inclinaison ou par le bas.

Vacuum Casting Apparatus 125/III

United States. A vacuum furnace for semi-continuous operation must satisfy the following requirements : (*a*) Oxidation of the residue in the hot crucible exposed to air between two successive runs should be minimised, (*b*) thermal stresses in the crucible should be avoided. This can be achieved by keeping the melting chamber continuously under reduced pressure and keeping the crucible warm by arranging for a rapid succession of the melts. The chamber of the furnace described consists of a feeding, a melting and a casting section which can be evacuated jointly or individually. Thus, the furnace can be charged and the ingot can be removed whilst melting is in progress. The raw metal is placed in a bucket in the feeding section. The feeding section is evacuated and the bucket moved by means operated from outside to a position above the crucible in the centre of the melting chamber. The bucket is tilted, the contents dumped into the crucible and the bucket returned to the feeding section. The feeding section is shut off from the melting chamber and the graphite crucible is heated up (by high frequency dielectric heating). For pouring, the crucible is tilted feeding the molten metal

Vol 3 195

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

to a trough which connects the crucible with the mould in the third section. The trough is heated to prevent chilling of the molten metal during transit and the mould is water-cooled. Radiation shields are placed in suitable positions. The furnace chamber, double-walled to provide for water-cooling, is made from stainless steel, the trough is preferably made from magnesia and the crucible from graphite. The vacuum should be of the order of 0.01 mm. Hg. Under normal operating conditions the furnace requires a fresh charge every 3–5 minutes.

Sommaire : Four à vide pour opération semi-continue.

J. H. Moore & National Research Corp. *U.S. Pat.* 2,625,719

Improvements i.o.r.t. Devices for Melting and Casting Materials under Air-Exclusion 126/III

Liechenstein. The vacuum furnace described consists of a single vacuum chamber, double-walled to facilitate water-cooling, containing all feeding, melting and casting assemblies. The crucible surrounded by a high frequency coil, is positioned near the centre of the chamber. The crucible-coil unit is fixed to a horizontal shaft which can be rotated from outside in order to tilt the crucible. The molten metal is poured directly into a mould fixed to the bottom of the chamber. An observation window and the feeding device are fitted to the top of the chamber. The latter can be closed air-tight by a hinged lid and consists of a multitude of segment-like compartments fixed in a horizontal position to a vertical shaft. The compartments are open at the bottom and rest on a horizontal plate which has an opening of the same size as one of the compartments. When the shaft is rotated the raw material placed in the various compartments will successively drop through the opening in the horizontal plate and fall onto a shoot located directly underneath which discharges the metal into the crucible.

Sommaire : Four à vide pour le même but que sous 125/III mais ayant des particularités de construction différentes.

A. Vogt *Brit. Pat.* 692,050

Arc-Melting of Non-Metallic Materials and a Redetermination of the Beryllia-Thoria System 127/III

See Abstract No. : 109/II

Determination of Oxygen in Chromium by the Vacuum Fusion Method 128/III

United States. A method for investigating the effects of operating temperature and metal bath composition upon the recovery of oxygen by the vacuum-fusion method is presented and exemplified by application to Cr. This method consists of using mixed powders containing metal and metal oxide, determining the O at various operating temperatures, and allowing the concentration of the sample metal to increase in the bath. The results are fitted by the method of least squares to an equation of the form :

$$R = a + c_1C + c_2C^2 + t_1T + t_2T^2 + kCT$$

where a, c_1, c_2, t_1, t_2 and k are the fitted constants, R is the recovery equal to 100 x (O found)/(O expected), C is the weight per cent. of chromium in the bath, and T is the operating temperature. For chromium, much better results were obtained by using a molten bath of 75% Fe and 25% Sn by weight than by using a bath of pure Fe. At 1,600°C the method with this type of bath gave about 85% recovery of O when no Cr was present. When the samples had increased the Cr to about 5% of the bath, the O recovery dropped to about 70%. The results were checked by modification of an acid solution method. Cr annealed at 800°C in vacuum was dissolved in 1.2N HCl. The insoluble residue was fused with potassium persulfate and potassium hydroxide in a nickel crucible. Dissolved in water, the fusion mixture gave a solution of potassium chromate in proportion to the undissolved chromic oxide. The latter was measured colorimetrically. Powders appeared to give better results with the vacuum fusion method when wrapped with Al foil and Fe wire rather than when placed in Ni capsules.

(*Nuclear Science Abstracts*)

Sommaire : Méthode d'investigation des effets de température et de la composition des bains sur le rétablissement de l'oxygène par la méthode de fusion sous vide.

Report by W. S. Horton & J. Brady *A.E.C. Rep.* No. KAPL. 874

38 — DISTILLATION — 38

Design and Use of an Electronic Pressure Controller 129/III

See Abstract No. : 122/II

Improvements i.o.r.t. Stills 130/III

United Kingdom. The design of a still is described which is intended for the production of drinking water from sea water in an emergency. The apparatus consists of two distillation systems, one working at atmospheric pressure and the other working at a slightly reduced pressure which is produced by a hand operated vacuum pump. The former consists essentially of a boiler heated by a burner for solid fuel. The water in the boiler is kept at a constant level by a suitable device. The steam collected at the top of the boiler is conveyed into a condenser. The condensate is passed to a collecting vessel *via* a heat exchanger. The condenser forms the link between the atmospheric and the low pressure system. The cooling medium is water, supplied from the feed water tank. As the space above the water is evacuated the heat liberated from the steam causes the water to

III — Vacuum Processing Techniques — III
Contd.

Abstract No. and References

boil. The pipe connecting the low pressure evaporator with a receiver tank passes through the feed water tank, where the steam is condensed. The distilled water is discharged to the aforementioned collecting vessel, as and when required. The vacuum pump is connected to the receiver tank. The low pressure system is operated intermittently. When the receiver tank is full, air is admitted and the water in the evaporator is returned to the feed water tank. Special provisions are made to remove the concentrated brine which collects in the evaporator. The heat exchanger mentioned before serves the purpose of pre-heating the water supplied to the atmospheric system. The apparatus facilitates the distillation of 9 parts of drinking water in the low-pressure system for every 10 parts distilled in the atmospheric system.

Sommaire : Un distillateur operant partiellement sous vide a été développé pour la production d'eau potable, partant d'eau de mer, en cas d'urgence.

L. R. B. Shackleton J. C. Wilson & National Research Development Corp. *Brit. Pat.* 691,789

39 — MISCELLANEOUS PROCESSES — 39

Improvements i.o.r.t. Vacuum Insulated Systems for Conveying or Storing Liquefied Gases 131/III

See Abstract No. : 126/II

Drilling by Electrons 132/III

United Kingdom. A new technique of drilling holes into materials such as tungsten, glass, porcelain, palladium, silver alloys and steel by electron bombardment in a high vacuum is reported. The holes are extremely small in diameter but can be several millimetres deep. An electron beam of 50 kV is used and its direction and concentration is controlled by means similar to those employed in electron microscopes. Drilling times vary from 1 to 20 seconds. A special technique is employed when drilling diamonds.

Sommaire : Brève note sur une nouvelle téchnique pour le perçage de petits trous par bombardment électronique.

Note by Anon. *World Sci. Rev.* 1, June 1953 40

Potash Refining in France 133/III

United Kingdom. Potash production of the mines in Alsace has risen in the last 6 years from 600,000 tons p.a. to 1,000,000 tons p.a. The crude potash-bearing ore mined contains 25–35% KC1, 55–65% NcC1 and 10% insoluble matter. The raw material is subjected to a series of crushing operations until it is ground to a screen size of –5 mm. In this form the material is marketed under the name of ' sylvinite ' as a straight fertiliser. But the greater part of the ground sylvinite is processed further with the object of separating the sodium chloride from the potassium chloride. Details of the refining process are given. It consists essentially of a series of heating and cooling operations. If a solution of the material saturated with sodium and potassium chlorides is heated to 230°F and then cooled, some of the potassium chloride precipitates. The resultant liquor, saturated with sodium chloride but not with potassium chloride is heated and used to dissolve the potassium chloride contained in the ground raw salts in a continuous dissolver. During the process the undissolved sodium chloride sinks to the bottom of the vat and is removed. The enriched liquor is clarified and fed to the vacuum cooler. Here the solution is cooled from 204°F to 107°F. The temperature drop causes the potassium chloride to crystallise. The liquor with the crystals in suspension is fed into settling tanks and the liquor drained off. The overflow from the tank is passed on to open-air vats where a second crystallisation of potassium chloride takes place. After drying in a cylindrical oven the crystals are ready for bagging and storage. Vacuum cooling was introduced in 1926. It is faster than the open-air process, facilitates the recovery of the heat of crystallisation, reduces the processing time from days to a few minutes and ensures a product of high purity.

Sommaire : La méthode employée pour la cristallisation et purification de potasse par un procédé sous vide est décrite.

Article by Anon. *Chem. Process Engng.* 34, April 1953 98, 99 & 102

Evaporative Cooling of Fruits and Vegetables 134/III

United States. In vacuum cooling the heat is removed from the article treated by evaporating some of the moisture it holds. If the pressure in the processing chamber is reduced, the water will boil at a lower temperature. In order to obtain a saturation temperature of 32°F the pressure has to be reduced to 4.6 mm. Hg. The smaller the pressure the larger is the volume occupied by the saturated water vapour given off by a certain quantity of water. This is demonstrated in the table reproduced below. Therefore large capacity vacuum pumps are

Table I. Properties of Water under Vacuum

Saturation Temperature, F	*Pressure, Inch of Mercury*	*Heat of Vaporisation, Btu per lb.*	*Volume of Saturated Vapour, cu. ft. per lb.*
32	0.18	1,073	3,301
40	0.25	1,069	2,445
60	0.52	1,058	1,208
80	1.03	1,047	634
100	1.93	1,036	351
212	29.92	970	27

Vol 3 195

III — Vacuum Processing Techniques — III

Contd.

Abstract No. and References

required for the process. Lettuce is now vacuum cooled on a commercial scale. Single heads can be vacuum cooled from 77 to 32°F in 5 minutes. Large batches require 30–55 minutes depending on the initial temperature and size of the batch. The removal of moisture has been found to be equivalent to 3.4–4.7% of the original weight. This figure is higher than that obtained by cooling in refrigerated air, but no detrimental effects have been observed. The water seemed to have withdrawn from all parts of the head and not only from the outside leaves. Non-leafy vegetables have also been tested. For instance broccoli, a non-leafy vegetable, can be cooled from 65°F to 40°F in 15 minutes. If pre-moistened it can be cooled to 32°F in the same time, and the loss of moisture is limited to 1% of the initial weight. Potatoes were found to be unsuitable for this form of treatment. This may be due to their surface being comparatively small and the fact that the intercellular spaces of the tissues are not continuous. Therefore the evaporation of the moisture from within is stopped. Peeled potatoes give slightly better results. Removal of the skin improves these conditions, but the speed of cooling is not rapid enough for practical purposes. Apples of the golden delicious variety 3 inch. in diameter were vacuum cooled 79 to 50°F in 45 minutes. The loss of moisture was equivalent to 1.9% of the original weight. But the vacuum cooled apples showed serious damage after storage of one month at 40°F. It appears that the success of the method depends on the ratio of surface area to volume available in the commodity to be cooled. Lettuce, spinach, endive, escarole, parsley, brussel sprouts and cabbage are suitable because they have a very large area. Broccoli, sweet corn, asparagus, celery, leeks have less surface area and, therefore, are less suited. Apples and potatoes are difficult.

Article by D. H. Dewey *Refrig. Engng.* 60, Dec. 1952 1281-1283

O *Sommaire* : Rapport sur les avantages offerts par le refroidissement de fruits et légumes par évaporation donnant en même temps les désavantages de la méthode.

Process for Producing Milk Fat Concentrate and Butter

135/III

United States. Approximately 30–40% cream is pre-warmed to 90–110°F in a vat and then heated to approximately 135°F by direct mixture with steam at a pressure of 90–100 lb./sq. inch in a conduit. The cream is filtered, flash-pasteurised at 190–4°F for 10–15 sec., and directed into a vacuum steam pasteuriser under pressure of 25–26 inch. of Hg; the maximum temperature within the chamber is approximately 130–5°F. Steam under 90 lb. pressure is introduced into the bottom of the pasteuriser, and desired pressures and temperatures are maintained by withdrawing vapour or volatile gases through an outlet at the top by using a vacuum pump. The cream collects in the lower portion of the chamber and is drawn off by the pump and forced into a centrifuging separator. The mixture containing 80–90% milk fat is pumped to the mixing chamber where it is mixed with a controlled aqueous solution of NaCl, starter, flavours, etc. The resulting mixture containing at least 80% milk fat is conducted to a cooling stabilising unit where the temperature is reduced to 40 – 60°F. Finally the desired texture of the butter is imparted in a tempering unit, and the final product is forced through a conduit to a packaging machine.

(*Chemical Abstracts*)

H. C. Horneman, R. V. Hussong, S. N. Quam, B. W. Hammer & Cherry-Burrell Corp. *U.S. Pat.* 2,630,388

Sommaire : Procédé de production de graisse de lait concentrée et de beurre faisant appel à le pasteurisation par vide et vapeur.

O

Vol. 3 1953

IV — Special Subsidiary Subjects — IV

Abstract No. and References

40 — GASES AND VAPOURS — 40

Supersonic Speeds in High Vacuum 38/IV

See Abstract No. : 91/II

Device for Preparing Gas Mixtures for Calibration 39/IV

United States. An apparatus has been developed for the preparation of calibrating gas mixtures mainly of interest in the field of infra-red spectroscopy. It consists of two 48 mm. internal diameter glass tubes, A and B, in a horizontal position each containing a loose piston and joined by a piece of tubing, carrying a glass stopcock D. The outer ends of the tubes carry a 2-way stopcock, E and F. In one position these stopcocks connect to atmosphere, in the other to a glass tube linking the outer ends of A and B. Binary gas mixtures are prepared as follows : Evacuate the apparatus, close all stopcocks, fill one tube with one gas at the desired pressure, fill the other tube with the other gas at the desired pressure. Open stopcock D and change the position of E and F to open the connection between A and B, then rock the apparatus allowing the loose pistons to slide backwards and forwards in the tubes. The composition of the mixture may then be calculated by using the formula :—

$$\text{Vol. \% A} = \frac{\text{Pressure in A x Vol. in A}}{\text{Pressure in A x Vol. in A} + \text{Pressure in B x Vol. in B}}$$

Note by A. Opler & E. S. Smith *Analyt. Chem.* **25**, April 1953 686

Sommaire : Appareil permettant le mélange précis de gaz.

Adsorption by Evaporated Copper Films at 78°K. Part I. Krypton and Hydrogen 40/IV

Canada. In the past work concerned with the sorption of hydrogen by a copper surface was carried out on surfaces produced by reduction of the oxide. The results disagree with those of Beeck who found that evaporated copper films do not adsorb hydrogen. The authors report on a study of hydrogen adsorption by pure evaporated copper films and films containing chemisorbed oxygen and bulk oxide. Nickel films containing 0.8% copper were also investigated. The adsorption was measured by a volumetric method at pressures below 2 mm. Hg. Details of the (vacuum evaporation) method employed for the deposition of the films are given. All but two copper films were annealed after deposition (with the vacuum cell at −78°K) by heating them to +25°C and cooling them to −78°K for 4 cycles in 30 minutes. The nickel films were evaporated with the chamber at 0°C and not annealed. All pressure measurements were corrected for the Knudsen effect resulting from the thermomolecular pressure by a method described in detail in an appendix to the publication quoted in Abstract No. 41/IV. Sorption was carried out in a liquid nitrogen bath with a precision better than 0.5×10^{-3} cm.³ S.T.P. The surface areas of all films were measured by krypton adsorption but in order to avoid contamination by krypton the hydrogen and oxygen experiments were carried out first. The B.E.T. method was used to determine the amount of gas needed to form a monolayer. The accuracy of surface area measurement was ±20 cm.². The necessary corrections taking into account krypton adsorption on the walls of the apparatus have been made. In order to calculate the area of the film from V_m values, assumptions for the packing and effective cross section of the adsorbed krypton atoms had to be made. The value of 14.6 Å² for the effective cross section of an adsorbed krypton atom (equal the mean of the values obtained from the liquid and solid densities) was used in this investigation in order to conform with Beeck's treatment. A table giving the summarised data for krypton and hydrogen adsorption on all films is partly reproduced below.

Table I Adsorption of Krypton and Hydrogen by Cooper

Surface					*Krypton Adsorption*			*Hydrogen Adsorption**	
Expt. No.	*Metal*	*State of Surface*	*Annealing†*	*Film wt. mg.*	*Vm Total Film* 10^{-3} *cm.³ s.t.p.*	*area* $m^2\ g^{-1}$	*Total Area of Film cm.²*	10^{-3} *cm.³ s.t.p.*	*pm* 10^{-3} *mm.*
6	Cu	Clean	n	27.1	230.0	33.2	9,010	5.3	2,017
12	Cu	Clean	a	212.2	16.4	0.303	643	3.1	1,343
11a	Cu	Oxygen	a	179.0	21.9	0.479	859	3.0	845
11b	Cu	Oxid. at O°C	a	179.0	21.9	0.479	859	0± 1	624
4	Ni + Cu	Clean	n	120.5	785.0	25.6	30,800	159.1	2,981

* at maximum pressure pm of experiment. † a, annealed; n, not annealed.

In order to obtain reliable data on hydrogen adsorption the packing of the hydrogen molecules had to be considered. It was assumed that 1 hydrogen molecule occupies 6.5 Å² in the case of physical adsorption and, when dissociated and chemisorbed as atoms, 14.2 Å² on copper and 13.4 Å² on nickel. The table shows that

the amounts of hydrogen adsorbed by copper is very small compared with krypton adsorption in all cases. The coverage on clean copper is about 1% at 78°K and 1.5 mm. Hg pressure. The presence of impurities does not increase the adsorption of hydrogen. Comparing the isotherms on clean copper with the adsorption of hydrogen on a pure nickel film or a nickel-copper film it is concluded that the hydrogen adsorption on copper at 78°K must be physical.

Sommaire : Recherche sur les propriétés d'adsorption de films de cuivre évaporés à 78°K concernant le krypton et l'hydrogène.

Article by G. L. Kington & J. M. Holmes *Trans. Faraday Soc.* **49**, 1953 417-423

Adsorption by Evaporated Copper Films at 78°K. Part 2. Krypton and Oxygen 41/IV

Canada. The interaction of oxygen with copper is of importance in connection with heterogeneous catalysis. As in the investigation quoted in Abstract No. 40/IV krypton was used to determine the area of the film. As before the measurements have been corrected for thermomolecular pressure. An experimental investigation into this effect is reported in an appendix. Apparatus, technique of film deposition and measurement method were the same as those described in the publication quoted in Abstract No. 40/IV. The experiments with oxygen consisted of a series of adsorption and desorption cycles. Details of the procedure are given. The cross section of krypton was taken to be 14.6 Å²; the cross section of oxygen was taken to be 13.6 Å² in the case of molecular adsorption based on the liquid density for oxygen at 78°K, and in the case of dissociation and ionisation a complete layer of oxide ions was estimated to require 5.25×10^{-3} cm.³ S.T.P. of oxygen per 100 mg. of copper. The first two increments of oxygen admitted to a fresh film at 78°K in the first cycle were completely adsorbed at pressures below 10^{-4} mm. Hg, whereas the following increments resulted in a much larger and measurable pressure. This did not repeat itself in subsequent cycles. Thus it can be assumed that the gas initially adsorbed was not desorbed by the pumping between cycles *i.e.* chemisorption must have taken place. This chemisorbed oxygen, if it is assumed that it was dissociated and ionised, represented 1.9 layers of oxide ions at an equilibrium pressure below 10^{-4} mm. Hg and 78°K. This was equivalent to a coverage of approximately $\frac{3}{4}$ of the surface. It was found that all oxygen admitted subsequently to the first two increments during the first cycle showed a marked hysteresis on desorption and, in addition, the amount adsorbed at any given pressure decreased each time. As krypton adsorption was completely reversible the hysteresis could not be due to capillary action. Considering both the phenomena observed it appeared that either the amount of surface available for physical adsorption or the activity of the surface to chemisorption was reduced. This seemed to point to a condition where oxygen was adsorbed by two different mechanisms simultaneously. When pumping was resumed one form of adsorption was reversed but not the other. A mixed phase established itself, where the amount of oxygen adsorbed by the non-reversible mechanism appeared to depend on the pressure. The authors compare their results with those of Arnold who studied oxygen adsorption on titanium dioxide at 78°K and come to the conclusion that the reversible adsorption noticed after the initial chemisorbed layer had formed at a pressure below 10^{-4} mm. Hg, is physical adsorption of molecular oxygen on that layer, whereas the ' strong ' adsorption noticed during the same phase of the experiment can only be considered to be a continuation of the chemisorption occurring below 10^{-4} mm. Hg in the absence of knowledge of any other mechanism.

Sommaire : Recherche sur les propriétés d'adsorption de films de cuivre évaporés à 78°K concernant le krypton et l'oxygène.

Article by G. L. Kington & J. M. Holmes *Trans. Faraday Soc.* **49**, 1953 425-432

A Miniature Helium Liquefier-Cryostat of Cascade Type 42/IV

See Abstract No. : 125/II

Helium Diffusion Through Glass 43/IV

United States. The permeation of helium through various types of glass has been investigated with the help of a mass spectrometer. The helium was filled into spheres of the glass to be examined at a pressure of 450 mm. Hg. The spheres had a diameter of 0.45 inch and a wall thickness varying from 0.145 to 0.203 mm. Hg. The wall thickness was checked by various electrical methods, described in detail, with an accuracy of ± 10%. The spheres were placed in a glass container of 25 cm³ volume with thick walls so that loss of helium to the outside could be neglected. The container could be evacuated and could be connected to a subsidiary collecting vessel or directly to the mass spectrometer. The container was surrounded by a furnace or by a low temperature bath, as required. Three methods were used to determine the rate of permeation. If gas penetrated through the wall of the spheres at a high rate the gas was transferred to the subsidiary collection system. From the period of collection, temperature and pressure the amount permeated could be computed. To verify that the gas measured was helium a small portion was analysed in the mass spectrometer. In the second method the gas was fed directly into the mass spectrometer, while the latter was set to record peak of mass 4 (helium) continuously. In the third method the outer container was used as the collecting vessel. After a given time the container was connected to the mass spectrometer and the resultant pulse, recorded in the instrument, was found to be proportional to the amount collected. For the case of rare gases and assuming non-dissociation and uniform temperature, permeation can be expressed by the formula : $q = KAt\,(p_1 - p_2)/d$, where K is the permeation velocity, A the area of the glass wall, t the time, p_1 and p_2 the gas pressure on the high and low side respectively and d the thickness of the wall. K is expressed in cm³. of gas at normal temperature and pressure (760 mm.

Vol. 3 1953

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

Hg) per sec. per cm.² area per mm. thickness per cm. Hg. The values of K for helium obtained in the experiments are given in a table partly reproduced below. It has been found that the lower the percentage of glass-formers present, the lower is the permeation velocity. Glassformers are SiO_2 B_2O_3 P_2O_5. An attempt to measure the permeation through mica which contains no glassformers showed that permeation is practically nil. The atomic arrangement of fused silica is shown diagrammatically indicating large holes which may promote permeation. If sodium or ion, the typical constituents of soda glass, are added, the holes are plugged with a consequent reduction of the permeation rate. The following conclusions are drawn : In the case of crystalline quartz, helium permeation is slow because of the compact structure of the material. In the case of silica glass, helium permeation is rapid because of the openings in the structure of the material. By adding non-glassformers such as sodium or lead the openings can be blocked and permeation reduced.

Full List of Glasses Investigated

No.	*Type of Gases*	*Manufacturers*
1	Fused SiO_2	General Electric Company
2	Vycor Brand	Corning Glass Works
3	Chemical Pyrex Brand No. 7740	Corning Glass Works
4	Borosilicate No. 650	Owens-Illinois Glass Company
5	Phosphate Glass. Only traces of SiO_2 are present in this hydrofluoric acid-resistance glass	Haverford Glass Company
6	Soda-Lime No. 0080	Corning Glass Works
7	Combustion or Ignition Tubing No. 1720	Corning Glass Works
8	X-ray Shield Glass	Pittsburg Place Glass Company
9	Lead Borate (78.2% PbO, molecular ratio $PbO/B_2O_3=1.12$)	General Electric Company

Abbreviated Table of the Values of Permeation Constant K Determined in the Experiments*

Temp. (°C)	*Permeation Constant K**
	Vycor Brand (2) (Glassformers 99%)
−78	2×10^{-12}
0	6.6×10^{-11}
26	1.6×10^{-10}
	SiO_2 Glass (Clear Fused Silica) (1) (Glassformers 100%)
−78	1.3×10^{-12}
−23	2.15×10^{-11}
0	5.3×10^{-11}
25	1.14×10^{-10}
	Chemical Pyrex Brand No. 7740 (3) (Glassformers 94%)
−78	4.9×10^{-14}
−23	1.6×10^{-12}
0	4.0×10^{-12}
25	9.1×10^{-12}
	Borosilicate No. 650 (4) (Glassformers 90%)
c23	9.4×10^{-14}
+25	9.2×10^{-13}
178	7.0×10^{-11}
	Phosphate Glass (5) (Glassformers 82%)
28	$1\,9 \times 10^{-14}$
	Soda-Lime No. 0080 (6)
25	7.5×10^{-15}

* *For dimension see text.*

Sommaire : La permeabilité de verres de différentes compositions du hélium à diverses températures a été étudiée.

Article by F. J. Norton *J. Amer. Ceramic Soc.* **36**, March 1953 90-96

Calculation of the Rate of Diffusion of Hydrogen Through a Plate of Palladium

44/IV

France. The work was undertaken in connection with a high frequency hydrogen ion source. The relation between the mass of gas diffusing through a plate of 1 mm. thickness with a pressure of 760 mm. Hg on one side and a vacuum on the other side and the temperature of the plate are derived. This calculation is based on the usual experimental and theoretical relation by which the mass of gas diffusing through a plate is proportional to the square root of the pressure and exp. $-E_0/2RT$ where E_0 is the heat of diffusion. The resulting relation contains two constants, the values of which were found by several authors but showed big differences. From these various constants as listed by Dushman the diffusion rate was tabulated and plotted in graphs for

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

different temperatures (at 50°K intervals) over the range 300°K to 1,000°K. After this the range of pressure 1 to 5 atmospheres was also considered. These tables and graphs are used to work out the diffusion rate obtainable from a practical palladium osmosis tube. The correction to the fundamental formula for the case of pressures less than atmosphere is also discussed and investigated numerically.

Sommaire : La vitesse de penétration de l'hydrogène à travers le palladium a été déterminée point par point entre 300 et 1,000°K et pour des pressions jusqu'à 5 atmospheres.

Article by E. Thomas *Soc. Roy. Belge Ing. Industr.* No. 1, 1953 19-32

Increased Sensitivity of Leak Detection with Hydrogen 45/IV

See Abstract No. : 123/II

Direct Determination of Oxygen in Rubber 46/IV

See Abstract No. : 176/I

41 — FREEZE DRYING — 41

The Sublimation of Ice in Vacuo 47/IV

See Abstract No. : 142/I

Freeze Drying in Toxicology 48/IV

Italy. The author summarises the theoretical fundaments of the ' freeze drying ' method which consists in drying a substance through sublimation of its solvent under high vacuum pressure. He then explains how this method is generally used in biology, and after describing the inventory and plant of a small laboratory, he reports some tests carried out in the field of chemical-toxicological analysis regarding the preservation of substances containing acid-phenylethyl-barbituric, aconitine, atropine, cocaine, emetine, physostigmine, morphine. The author finally points out, as a result of these tests, that substances which have been desiccated in the above-mentioned way and kept at room temperature, preserve their original quantity for over a year. Hence this method facilitates considerably the task of the toxicological analyst.

(*Author*)

Sommaire : L'auteur discute les emplois de la lyophilisation dans le domaine biologique et rend compte de tests dans le domaine de l'analyse toxicologique.

Article by G. Di Castro *Zacchia* 16, April-June 1953

Application of Vacuum Technique to the Production of Blood Plasma and Analogous Biological Products 49/IV

France. The progress recently made in the field of the chemistry of proteins was largely facilitated by the introduction of the freeze drying technique. Proteins are micromolecular substances consisting of a large number of amino-acids and have a molecular weight ranging from a few thousand to several million. The proteins differ from other macromolecules in two ways. They are formed from 20 to 25 different amino-acids which between themselves differ by their radicals, their molecular weight and their relative position in the molecule. In addition, the radicals contained in the protein molecule are chemically active in various directions. Thus, there exists an infinite variety of protein types. For instance the albumin protein molecule of bovine serum of the moderate molecular weight of 70,000 contains about 600 amino-acid molecules of 20 different kinds. It possesses 103 free-basic functions, 87 free-acid functions, 77 alcohol functions, a number of benzene nuclei and many other groups. Proteins play an important part in the life of all cells. Many chemical reagents normally not harmful to macromolecules may cause denaturation, but it has been found that freezing prohibits their denaturation. The author explains the basic principle of the freeze drying technique and proceeds to describe the design of various commercially available freeze drying apparatus. Two different methods of refrigeration are used : Flosdorf, Strumia, International Equipment, Usifroid and the French Centre of Blood Transfusion employ appafatus where the flasks are placed on rollers submerged in the refrigerant in a horizontal position and the British design provides for the flasks to be placed on a revolving support in a vertical position. The French firm Les Chantiers de la Loire have designed an apparatus where the liquid refrigerant is used for the double purpose of freezing the solution in the flasks and revolving the flasks. The drying plant available can also be divided into two groups. In one type a batch of flasks is placed into a chamber and the chamber is evacuated. In the other type the flasks are joined individually to a vacuum manifold. The author concludes with a description of the design of a freeze drying apparatus developed by himself and based on the apparatus by Wyckoff and Logsdin. The apparatus consists essentially of a cabinet containing a vacuum manifold with a condenser in the centre. The flasks are sealed individually to the outside of the manifold and are exposed to hot air circulated by means of a fan in the space between manifold and cabinet walls during the drying operation.

Sommaire : Revue générale de la chémie du sang et du plasma sanguin et différents procédés de lyophilisation du plasma.

Article by J. Lewin *Le Vide* 8, March 1953 1307-1319

Vol. 3 1953

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

The Histological Preparation of Human and Animal Tissues by Freeze Drying

50/IV

United Kingdom. The orthodox techniques of fixation, dehydration and embedding of tissues result in considerable modification of the tissue cell due to loss of certain soluble substances, reduction of the activity of the enzymes and displacement of certain tissue constituents. This effect is minimised by the use of the freeze drying technique. The tissue to be dried is cut into small pieces and frozen in isopentane, cooled down to –160°C with liquid oxygen. The frozen tissue is placed in the drying chamber and allowed to warm to –40°C. Then the chamber is evacuated and the moisture contained in the tissue removed by sublimation. Finally, in preparation of section cutting, the tissue is embedded under vacuum in paraffin wax of a temperature of +55°C. A simple apparatus costing no more than £100 to build is described. Two Pyrex glass tubes, 28 mm. wide and 120 mm. long, serving as drying chambers and fitted with stopcocks are sealed to a glass tube, 20 mm. wide and 350 mm. long, connecting with a condenser, in the form of a tube 40 mm. wide and 250 mm. long. The three vessels are cooled by Dewar flasks, 90 mm. wide and 200 mm. long, which can be removed when required and are fitted with an acetone and carbon dioxide solution in the case of the drying chambers and liquid nitrogen in the case of the condenser. A tube 20 mm. wide, 200 mm. long connects with a phosphorous pentoxide trap. The trap consists of a circular brass well closed at the top by a glass plate and contains 3 Pyrex Petri dishes arranged in tiers holding the drying agent. A brass tube, 20 mm. wide, leads to a McCleod Gauge and the vacuum pump. The latter is of the mechanical type, has a displacement rate of 48 l./min. and gives an ultimate pressure of 0.00005 mm. Hg. To determine the optimum temperature, at which the tissue should be held during sublimation is a matter of great complexity in the case of tissue drying and it is recommended to determine the required data by practical experiment. The author found that sublimation at –30°C gives good results; –40°C may give more consistent results but the drying rate is slower due to the lower vapour pressure of the ice at that temperature. Tissues possessing a maximum of surface seem to give the best results. Skin, intestinal mucosa and pancreas present no difficulties, liver and kidney present some difficulties and organs with tough capsules such as spleen, testis, etc. are extremely difficult to preserve. Various techniques for the manipulation and mounting of sections are mentioned. The staining of freeze-dried sections can be carried out by conventional methods in most cases. But it was found necessary to denature the proteins as yet unfixed before staining and impregnation. The author used absolute alcohol for the purpose. The behaviour of freeze-dried sections during treatment by various staining techniques is discussed. Details of one of them, the Heidenhain's azo-carmine technique, modified to suit freeze-dried sections are given and are reproduced below.

Method of Staining

1. Distilled water, 2–3 minutes.
2. 5 per cent. aqueous phosphotungstic acid, 2-3 hours.
3. Rinse in distilled water.
4. Stain in aniline-blue orange G mixture, 6–12 hours, made up as follows

Aniline blue	0.5	gm.
Orange G	2	gm.
Water, distilled	100	ml.
Acetic acid, glacial	8	ml.

5. Differentiate in absolute alcohol.
6. Clear in xylol.
7. Mount in either balsam or D.P.X.

Sommaire : Le domaine de la lyophilisation de tissus est passé en revue et un appareil convenable décrit et des conseils donnés quant à la coloration des specimens sechés.

Article by L. J. Wright *J. Med. Lab. Tech.* 11, April 1953 73-86

Storage of Normal Tissue Grafts

51/IV

See Abstract No. : 175/I

The Breaking of Tobacco Mosaic Virus Using a New Freeze Drying Method

52/IV

United States. Tobacco mosaic virus (TMV) preparations examined in the electron microscope are known to contain a large number of particles of an approximate length of 3,000 Å and a small number of shorter particles. It is believed that the full length of the virus is 3,000 Å and Williams has, in fact, made preparations which contained almost exclusively particles of that length. No satisfactory explanation exists on the nature and origin of the smaller particles. The authors studied these conditions in a series of experiments. Three lots of virus of various concentrations and age were used. Details of the method of preparation are given. The spray freeze drying method originally described by Williams was employed but, whereas Williams arranged for the collodion film supporting the preparation specimen to be in direct contact with the cold metal block during the freezing stage, the authors sprayed the preparation onto a collodion film supported by a specimen screen. This eliminated the stripping operation, but freezing was slower. Specimens prepared at dry ice and liquid air temperature showed a large number of short particles lying in a line as if they were broken pieces from a single rod and adding up to approximately 3,200 Å in each case. Specimens prepared at normal refrigerator temperatures showed only a few broken particles. Calculating the length of the individual short pieces making up rods of a minimum total length of 1,500 Å gave a value of about 400 Å p. piece as an average. Only 2 short pieces out of a total making up 542 rods were shorter than 200 Å. Assuming that the short pieces come from a single virus particle a movement must have occurred during the breaking process as the broken

Vol 3 195

IV — Special Subsidiary Subjects — IV
Contd

Abstract No. and References

pieces are not in contact with each other. The order of the magnitude of this movement is indicated by the data shown in the table partly reproduced below which is based on the measurement of 10 broken rods and 10

Representative Dimensions of Broken and Intact Tobacco Mosaic Virus Particles

Number of broken pieces	*Distance between far ends (D)*	*Sum of lengths (L)*	*Ratio D/L*	*Unbroken particle lengths*
8	5,220 Å	3,300 Å	1.55	2,600 Å
9	4,760	3,340	1.42	3,280
6	4,590	3,290	1.39	2,900
7	4,150	3,120	1.33	3,300
8	5,740	3,280	1.75	3,060

unbroken rods, selected at random in both cases. The lengths of the unbroken particles are nearly the same as the sums of the lengths of the broken particles but the distances between the far ends differ. The cause for the breakages is still obscure. Only one fact has been established, *i.e.*, the breakages occur only at very low temperatures. It may be that the particles contract during the freezing and drying operation and break in places, where they have frozen onto the substrate. Thermal expansion or contraction alone cannot be the cause as the movement is too large. Another possible explanation is that the rods are shattered when hitting the support during spraying.

Article by R. V. Rice, P. Kaesberg & M. A. Stahmann *Biochim. et Biophys. Acta* 11, 1953 337-343

Sommaire : La méthode de lyophilisation de Williams modifiée a été utilisée pour l'étude des dimensions du virus mosaique du tabac.

Recovery of Caprinised and Lapinised Rinderpest Viruses from Condensed Water Vapour Removed During Desiccation

53/IV

Nigeria. Processing material containing Newcastle disease virus Stein and Rogers found virus in the water condensate removed from the machine after use. In order to investigate the position tests were carried out in a centrifugal freeze dryer, which was to be used for the desiccation of materials containing virulent rinderpest. As the apparatus was normally used for the desiccation of caprinised and lapinised rinderpest virus vaccines, virus of the same type was employed in the experiments. Animals inoculated with 5 ml. of condensate, collected after 10 hours processing of the materials showed the typical symptoms confirming the presence of the virus in the condensate. It would appear that the greater part of the virus found in the condensate is carried out during the early stages of the freeze drying operation. Swabbing the walls of the vacuum chamber after processing gave negative results. It is emphasised that the machine should be disinfected, and the condensed water vapour collected in a disinfectant.

Letter by A. L. C. Thorne *Nature* 171, 4.4.53 609

Sommaire : La question de la sterilité du lyophiliseur après l'emploi, a été examinée expérimentalement.

45 — ELECTRONICS — 45

A Portable 250-Kilovolt Accelerator

54/IV

United States. An air-insulated linear accelerator has been developed which can be moved from place to place and consists of several units fitted with castors as follows : (1) The power supply (a 250 kV Cookroft Walton Generator), (2) the high voltage terminal incorporating the ion source and all necessary controls, (3) the accelerator system and (4) the pumping system. The generator supplies 5 mA at 250 kV. The high voltage is passed to the accelerator terminal through a shielded cable. The circuit is a half-wave voltage quadrupler operating at 400 c.p.s. A polished aluminium cabinet containing the ion source and controls serves as the high voltage terminal. The cabinet is mounted on 4 Textolite columns, 5 inch. outer diameter and 27 inch. long, fitted to a steel base. The ion source is supplied by a Kovar 115-volt, 1-kva 400 c.p.s. permanent magnet alternator incorporated in the unit. The motor driving the alternator is fixed to the steel base and the belt drive passes through one of the supporting columns. The accelerator unit consisting of accelerator tube, drift tube and target assembly is placed on a dolly in a horizontal position. This facilitates joining unit (2) and (3) in such a manner that the ion source fits into the terminal through a large grommet. Details of the accelerator tube are given and the design of the magnetic-type ion source used is shown in a sketch. The vacuum system consists of a 6 inch. oil diffusion pump, an oil booster pump and a mechanical fore pump. The operational pressure is 3×10^{-6} mm. Hg. The plant has been used to obtain neutrons from tritium absorbed in zirconium by the T^3 (d, n) He^4 reaction. Employing a deuteron beam of 300 μ A at 250 kV more than 10^{10} neutrons per second were obtained with fresh targets. About 10^9 neutrons per second could be obtained over a period of several hours.

Article by T. A. Bergstrahl, K. L. Dunning, E. Durand, C. H. Ellson, K. H. Howerton & W. Slavin *Rev. Sci. Instrum.* 24, June 1953 417-419

Sommaire : Description d'un accelerateur de 250 kV portable.

The Electronic Clean-Up of Gases in Sealed-Off Vacuum Systems

55/IV

See Abstract No. 134/I

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

Field Emission : Large Current Densities, Space Charge and the Vacuum Arc

56/IV

United States. Field emission was obtained from a single crystal W emitter under conditions of very high vacuum and clean surfaces. The geometry of the emitter was determined by electron microscopy. Certain conclusions were drawn as a result of these experiments. It is thought that current density was the dominant criterion for the initiation of the vacuum arc.

(*B.S.I.R.A. Bulletin*)

Sommaire : Le mécanisme de l'emission de champ avec reférence particulière aux déclenchements de l'arc sous vide a été étudié.

Article by W. P. Dyke & J. K. Trolan *Phys. Rev.* **89**, 15.2.53 799-808

A Simple Varying Capacitor Method for the Measurement of Contact Potential Difference in High Vacuum

57/IV

United Kingdom. The measurement of the contact potential difference between two metal surfaces facilitates the determination of the work function of a metal as the contact potential difference is numerically equal to the difference of their work functions. Measurement of contact potential difference can be effected in two ways: (*a*) The thermionic emission characteristics of the materials are investigated involving the use of an electron beam or (*b*) the photoelectric properties of the materials are studied. It has been shown in the past that a polycrystalline metal film presents a surface comprised of patches of different work function. This patchiness of the surface places a limitation on the usefulness of both these methods. The only method, by which true average contact potential difference between the surfaces is measured is that of Kelvin. In this method the metals under investigation form the active surfaces of a condenser. If a contact potential difference exists, the capacity of the condenser changes and a flow of charge results. By arranging for one of the plates to vibrate the flow of charge can be made to oscillate and a signal is obtained which can be amplified and registered on a cathode-ray oscilloscope. If the signal is reduced to zero by applying an external potential in series with the condenser this potential is equal to the contact potential difference. The author describes a refined form of the Kelvin apparatus for use at every low pressures. Details of the improved design are given. The condenser consisted of two circular tungsten plates, 2 cm. diameter and 0.0025 inch thick, facing each other. Both plates were joined to, and suspended from, the two central wires of a 10-wire valve-pinch. One of the plates was moveable and, by tilting the pinch, could be separated from the other by about 2 cm. The metals to be examined were deposited on the tungsten plates by evaporation. Molybdenum wires serving as evaporation sources were placed in front of two openings in a molybdenum cage surrounding the plates as a shield against stray electrostatic charges. Special safeguards were taken to prevent the metal vapour reaching the wrong plate. The whole assembly, including all parts essential for the measurement and gettering, was connected to the remaining leads in the valve-pinch, and sealed into a tube envelope of 3 inch. diameter and 7 inch. length. The envelope was evacuated to a pressure of $3–5\times10^{-8}$ mm. Hg by a mercury diffusion pump unit, fitted with a liquid air trap between valve and vapour pump and a second liquid-air trap in the backing line. The required vibration of the fixed plate was effected by tapping the glass envelope. The condenser was placed in series with a potentiometer, the whole being shunted by an 8–MΩ high stability carbon resistor. Details of the circuitry and an estimate of the signal voltage developed across the resistor are given. The contact potential difference between copper (–) and silver (+) films determined by the method described, was 0.285 ± 0.03 V. This value agrees with that obtained by Mitchell and Mitchell with the electron beam method. The author, comparing his results with those of other workers comes to the conclusion that the value of the work function depends on the structure of the surface investigated.

Sommaire : Forme raffinée de la méthode de Kelvin pour la détermination de la valeur de la travail de sortie d'un métal.

Article by H. P. Myers *Proc. Phys. Soc.* **66B**, June 1953 493-499

Work Functions of Gas-Coated Tungsten and Silver Surfaces

58/IV

United States. Gas discharge phenomena are known to depend on the work functions of the electrode materials. The measurements in this paper refer to the value of the work function of tungsten and silver after exposure to a glow discharge in hydrogen, nitrogen, oxygen, Freon 12, argon, helium and water vapour. Measurements were carried out with a modification of the Oatley magnetron contact potential method. The magnetron action of an axial magnetic field upon the electron current from the tungsten filament reference surface to the cylindrical diode collector has been made use of. The anode consists of the metal or treated surface whose contact potential difference, with respect to the tungsten filament, is to be found. The magnetron assembly was mounted in a Pyrex tube, pumped by an oil diffusion pump. The collector assembly could be moved magnetically from the measuring position (*down*) to a surface treatment position (*up*). In the latter position three auxiliary filaments served for electron bombardment or evaporation of a clean silver surface on the inside, or for ion bombardment. A series of measurements on clean and treated surfaces was made. The results for the tungsten surface were obtained accidentally when the tungsten filament of the magnetron burnt out several times during high temperature flashing. The largest changes 5.8 and 4.2 ev from the clean 4.67 ev value were obtained when operating the tungsten surface in oxygen and hydrogen glow discharges respectively. Silver changed from an average value of 4.21 ev to about 3.8 ev after a hydrogen discharge and to 5.4 ev and 5.5 ev after an oxygen and Freen 12 discharge. Water vapour at 20 mm. Hg only had very little effect. The results are in general agreement with other work.

Sommaire : Les valeurs de travail de sortie du tungstène et de l'argent ont été mesurées après exposition à une décharge ionisante dans plusieurs gaz et de la vapeur d'eau.

Article by G. L. Weissler & T. N. Wilson *J. Appl. Phys.* **24**, April 1953 472-475

Vol 3 195

IV — Special Subsidiary Subjects — IV
Contd.

Abstract No. and References

47 — GLASS PROCESSING — 47

The Bonding of Siliceous and Metallic Components 59/IV

Germany. The design of electronic apparatus frequently requires air- and vacuum-tight bonding between metal and glass or ceramic components. The present article surveys the progress of the bonding technique as revealed in recent (German) Patent Applications. In the German Patent 851664 it is proposed to employ an intermediary layer of slightly oxidised aluminium or copper which will prevent uneven shrinking after the joint has been made. Drops of the molten material are sprayed on the metal surface after cleaning and adhesion to the glass is effected by a pressing operation or alternatively by causing the metal to diffuse into the glass surface during a heat treatment. A special technique of sealing metal conductors of the molybdenum foil type vacuum-tight into quartz glass is described in Patent No. 819432. The arrangement consists of a heater device combined with a small press. The quartz tube, with the conductor in it, is placed on a support and heated up first. When the appropriate temperature has been reached the support is rapidly removed into a position in the vertical axis between the die and the punch of the press. Under the action of the press the seal is effected by flattening the tube. Titanium hydride (TiH_4, TiH_2, Ti_2H_3) is known to give a coating which adheres firmly to a ceramic surface, if it is applied under vacuum. In Patent 842469 a method of joining metal to a refractory such as titanium dioxide is described which uses titanium hydride as the bonding agent. In a particular application the upper end of a cylinder of refractory material is coated with a thin layer of titanium hydride powder mixed with a nitro-cellulose lacquer as binder. A ring of silver- gold- or copper solder is embedded in the titanium hydride layer and brought into contact with the metal part to be joined to the cylinder. The whole assembly is placed in an oven and heated in vacuum or in the presence of hydrogen to about 800-1,200°C to effect soldering. Suitable refractories are titanium dioxide, zircon, porcelains, quartz, etc. Finally in Patent No. 847571 it is proposed to use a densely sintered ceramic mass containing more than 15% cristobalite as an intermediary layer to obtain stress-free joints between the more common metals used in the construction of apparatus such as iron, copper and brass. Due to the admixture of the cristobalite the expansion coefficient of the material can be varied to suit requirements within very wide limits, but as the cristobalite loses its expansion properties above 400°C, this method can only be used where soft soldering is still satisfactory.

Sommaire : Une série de brevets decrivant différentes méthodes pour obtenir des liaisons étanches à l'air et au vide entre des corps métalliques et due verre ou des céramiques, est présentée.

Article by F. Reinhart *Glas- Email- Ker. Tech.* **4**, April 1953 119-121 & **4**, May 1953 167-169

High Vacuum Glass-Kovar Melts 60/IV

Germany. Descriptions are given of glass-metal melts in general and of glass-Kovar in particular. The determination of stress between glass and metal, the composition of Kovar (54% Fe, 29% Ni and 17% Co) and related alloys, and the glazing of Kovar with the relevant expansion coefficients are then dealt with. Finally, the effects of electrolysis on glass, and the behaviour of Kovar with metallic coatings of high electrical conductivity are considered. Reasons are also included for the preference of lead-free glass. 12 illustrations and 6 references.

(*Science Abstracts*)

Sommaire : Description de joints métal-verre en général et Kovar-verre en particulière.

Article by W. Duesing *Telef. Ztg.* **26**, March 1953 111-20

Vol
3
195

Cumulative Author Index, Vol. III (1953)

(For Abstract Nos. 1/I-68/I; 1/II-40/II; 1/III-52/III; 1/IV-18/IV see January issue)
(For Abstract Nos. 69/I-131/I; 41/II-84/II; 53/III-96/III; 19/IV-37/IV see April issue)

Author	Title of Publication	Abstr. No.
Adams, P. T., *et al.*	Special Equipment for Carbon^{14} Work.	91/III
Aerocessoires Simmonds	Improvements r.t. Pressure-Gauges.	98/II
Agruss, M. S., *et al.*	Falling-Film Type of Vacuum Still with Rotating Partial Condenser.	87/III
Aitken, M. J.	An Electrical Analogue to a High Vacuum System.	148/I
Aitken, P. B.	Vacuum Sealing Irradiation Containers by Cold Welding.	18/I
Akkeren, L. A. van, *et al.*	The Semi-Continuous Deodorisation of Oils in All-Glass Equipment.	51/III
Alers, G. A., *et al.*	Increased Sensitivity of Leak Detection with Hydrogen.	123/II
Allen, K. W., *et al.*	The Recovery and Purification of Tritium and He^3 Used in Ion Accelerators.	23/IV
Allen, R. R., *et al.*	The Semi-Continuous Deodorisation of Oils in All-Glass Equipment.	51/III
Almqvist, E., *et al.*	The Recovery and Purification of Tritium and He^3 Used in Ion Accelerators.	23/IV
Anon.	Important Steps in Investment Casting.	63/I
Anon.	Industrial Uses of Vacuum.	3/I
Anon.	Isotopic Method Determines Water Content.	43/I
Anon.	Gas Content Control.	58/I
Anon.	15 MeV Linear Accelerator.	16/IV
Anon.	Vacuum Treatment of Fish after Precooking Removes Undesirable Moisture and Odours.	50/III
Anon.	Vacuum Dehydration of Streptomycin.	7/II
Anon.	Vacuum Cooling Saves Costly Process Time.	49/III
Anon.	Newly Developed Pilot-Plant Batch Deodoriser Has Many Uses in Vegetable Oil Research.	52/III
Anon.	Purifying Titanium Sponge by Vacuum Distillation.	38/III
Anon.	Vacuum Bakeout Ovens.	61/II
Anon.	Sorbed Gases and High Vacuum Systems.	79/I
Anon.	Powdered Mashed Potatoes.	95/III
Anon.	High Quality Plating in a Vacuum.	54/III
Anon.	Environmental Test Chamber	83/II
Anon.	Conference on High Vacuum Technique.	69/I
Anon.	Vacuum Deposition becomes a Trade.	53/III
Anon.	Determining Gas in Aluminium Melts.	184/I
Anon.	Drilling by Electrons	132/III
Anon.	High Vacuum Metallising of Metals and Plastics.	114/III
Anon.	Storage of Normal Tissue Grafts.	175/I
Anon.	Potash Refining in France.	133/III
Anon.	New Field for High Vacuums.	169/I
Anon.	Good Carpets from Good Cops.	146/I
Aprison, M. H., *et al.*	An Improved Historadiographic Apparatus.	99/II
Asao, S., *et al.*	A Large Unwelded Vacuum Gate Valve.	119/II
Auwaerter, M.	The Stability of Inorganic Compounds in a High Vacuum.	62/I
Auwaerter, M.	Improvement i.o.r.t. Electric Discharge Devices for the Measurement of High Vacuums.	97/II
Bachman, C. H., *et al.*	Positive and Negative Ions in Cathode-Ray Tubes.	153/I
Bacon, C. G., *et al.*	Diffusion Measuring Meter.	24/II
Bailey, D. L. R., *et al.*	Heat Transfer to Mercury.	30/II
Baldock, R., *et al.*	Mass Spectrometer Studies of High Vacuum Materials.	28/II
Barbet, Societé des Etablissements	Improvements in the Functioning of Continuous Vacuum Distillation or Rectification Apparatus.	86/III
Barsch, G., *et al.*	Radiation from Freshly Evaporated Layers of Metals and Salts.	48/I
Barthauer, G. L., *et al.*	Analysis of Synthesis Gas. Rapid and Highly Accurate Method.	21/IV
Barton, D. M.	Vacuum 'O'-Ring Seals.	72/II
Bateman, J. B., *et al.*	Preparation of Uniformly Dispersed Specimens of Particulate Matter for Electron Microscopy.	54/II
Bateson, S.	The Application of Aluminium Reflecting Films to Glass and Plastics by Thermal Evaporation. Part II.	55/III
Bayer Farbenfabriken, *et al.*	Vacuum Dryer for Drying of Temperature-Sensitive Materials Supplied in Paste Form.	34/III
Benedict, Q. E.	The Technique of Vacuum Still Operation.	43/III
Bennett, A. I., Jr.	Rapid Graphical Procedure for Oven Design.	80/I
Bennett, W., *et al.*	Preparation of a Tritiated Self-Luminescent Material.	112/I
Benzinger, H.	New Methods for the Determination of the Optical Constant of Metals.	29/I
Berger, R. W.	Improvements in the Simple Distillation of Fatty Acids by Continuous Methods.	45/III
Bergstrahl, T. A., *et al.*	A Portable 250-Kilovolt Accelerator.	54/IV
Bernstein, I. A., *et al.*	Preparation of a Tritiated Self-Luminescent Material.	112/I
Biram, J. G. S.	Resistance Heaters for Vacuum Evaporation.	99/III
Birmingham Small Arms Co. Ltd.	Improvements i.o.r.t. The Production of Coatings on Ferrous Articles.	123/I

Vol. 3 1953

Cumulative Author Index, Vol. III (1953)

Contd.

(For Abstract Nos. 1/I-68/I; 1/II-40/II; 1/III-52/III; 1/IV-18/IV see January issue)
(For Abstract Nos. 69II-131/I; 41/II-84/II; 53/III-96/III; 19/IV-37/IV see April issue)

Author	Title of Publication	Abstr. No.
Black, J. H., *et al.*	Vacuum Pumping Apparatus.	88/II
Blaw-Knox Co., *et al.*	De-airating and De-oiling Citrus Juices.	93/III
Bliss, H., *et al.*	Rectification at Reduced Pressures.	44/III
Bloomer, R. N., *et al.*	The Electronic Clean-Up of Gases in Sealed-Off Vacuum Systems.	134/I
Bobenrieth, A.	A Degassable Penning Gauge.	51/II
Bockris, J. O'M, *et al.*	The Solubility of Oxygen in Gamma Iron.	125/I
Boettcher, A.	Glass Surfaces in High Vacuum.	23/II
Boettcher, A.	Research into Metals.	180/I
Bonnel, H., *et al.*	The Assessment of the Quality of Zirconium Powder Intended for Use in the Production of Vacuum Tubes.	76/I
Bozman, W. R., *et al.*	Electrodes Metal-Halide Lamps.	155/I
Bradley, D. E.	A New Method of Making Electron Microscope Specimen Support Films.	106/II
Brady, J., *et al.*	Determination of Oxygen in Chromium by the Vacuum Fusion Method.	128/III
Brasunas, A. deS.	Sub-Surface Porosity Developed in Sound Metal During High Temperature Corrosion.	61/I
Braybrook, A., *et al.*	Improvements i.o.r.t. the Production of Coatings on Ferrous Articles.	123/I
Breckenridge, R. G., *et al.*	Titanium Dioxide Rectifiers.	15/I
Brewer, L.	Recent Determinations of the Vapour Pressure of Graphite.	35/I
Briggs, L. J.	The Limiting Negative Pressure of Mercury in Pyrex Glass.	93/II
British Oxygen Co.., *et al*	Improvements i.o.r.t. Vacuum Insulated Systems for Conveying or Storing Liquefied Gases.	126/II
British Thomson-Houston Co. Ltd., The	Improvements i.o.r.t. Metal Vaporisers.	61/III
Broder, J., *et al.*	Evaporation of Germanium Films from a Carbon Crucible.	100/III
Broken Hill Associated Smelters Proprietary Ltd., *et al.*	Distillation of Metals.	37/III
Brown, R. W.	Vacuum Metallising of Metals and Plastics.	2/III
Brown, S. C., *et al.*	An All-Metal Vacuum Valve.	35/II
Bruce, E. W.	The Sublimation of Ice in Vacuo.	142/I
Bunt, E. A., *et al.*	The Design of High Vacuum Systems. Part I & II.	71/I
Burgerjon, J. J., *et al.*	The Synchrocyclotron at Amsterdam. IV. Details of Construction and Ancillary Equipment.	37/IV
Burns Brown, W., *et al.*	Behaviour of Fumigants During Vacuum Fumigation. I. Penetration of Methyl Bromide into Boxes of Dates.	73/III
Busbridge, W. G.	A New Design of Filter for Dry-Boxes. Type D/147.	46/II
Bush, W. E.	Causes of Failure of Vacuum Systems.	5/I
Bush, W. E.	Design of Radiation-Heated Cathodes for Ion Pumps.	2/II
California Research and Development Co., *et al.*	Development of a Large, Linear Jet, Mercury Diffusion Pump Having High Pumping Speeds in the 10^{-6} mm. Mercury Absolute Pressure Range.	41/II
Cantwell, J. B.	Impregnation Improves Casting Quality.	32/III
Carr, G. W.	Economical Finishing with Vacuum Metallising.	4/III
Castro, G. di	Freeze-Drying in Toxicology.	48/IV
Catlin, J. R., *et al.*	A Single Air Ejector as a Suction Motor for Dry Boxes.	45/II
Caule, E. J.	A Novel High Vacuum Furnace.	40/III
Cherry-Burrell Corp., *et al.*	Process for Producing Milk Fat Concentrate and Butter.	135/III
Chester, P. F., *et al.*	A Miniature Helium Liquefier-Cryostat of Cascade Type.	125/II
Christian, S. M., *et al.*	The Use of Radioactive Materials for the Generation of High Voltage.	14/I
Clark, Jr., L. C., *et al.*	A Large Capacity, All-Glass Dispersion Oxygenator and Pump.	41/I
Clemmons, J. J., *et al.*	An Improved Historadiographic Apparatus.	99/II
Cole, D. G., *et al.*	A Powder Camera Modification for High Temperature Photographs.	64/I
Conn, G. K. T., *et al.*	Cold Cathode Ionisation Gauges for the Measurement of Low Pressures.	50/II
Cooper, J. N., *et al.*	On the Preparation of Magnesium Targets from MgO.	47/III
Cope, W. F., *et al.*	Heat Transfer to Mercury.	30/II
Corliss, C. H., *et al.*	Electrodeless Metal-Halide Lamps.	155/I
Coward, L., *et al.*	A Single Air Ejector as a Suction Motor for Dry Boxes.	45/II
Coyle, J. E., *et al.*	An All-Metal Vacuum Valve.	35/II
Crites, G. J.	Vacuum Furnaces for Melting or Processing of Metals.	121/III
Croft, A. J.	A Big Expansion Helium Liquefier.	39/II
Daglish, H. N., *et al.*	Cold Cathode Ionisation Gauges for the Measurement of Low Pressures.	50/II
Dardel, G. von,	Combined Pirani and Ionisation Gauge Circuit.	96/II
Dargent, R., *et al.*	Aluminium Films in Electron Microscopy: The Production of Replicas with a Great Resolving Power.	105/II
Darling, D., *et al.*	A Single Air Ejector as a Suction Motor for Dry Boxes.	45/II
Davey, T. R. A., *et al.*	Distillation of Metals.	37/III
David, J. P., *et al.*	The Influence of the Electrical Field on the Performance of Thin Films.	21/III
Dawson, I. M.	Techniques for the Electron Microscopy of Crystals.	104/II
Dennard, F., *et al.*	A High Temperature Resistance-Heated High Vacuum Furnace.	123/III

Cumulative Author Index, Vol. III (1953)

Contd.

(For Abstract Nos. 1/I-68/I; 1/II-40/II; 1/III-52/III; 1/IV-18/IV see January issue)
(For Abstract Nos. 69/I-131/I; 41/II-84/II; 53/III-96/III; 19/IV-37/IV see April issue)

Author	Title of Publication	Abstr. No
Department of Scientific and Industrial Research.	Bibliography of Industrial Drying.	83/I
Department of Scientific and Industrial Research.	Study of Vacuum Fumigation Techniques.	72/III
Department of Scientific and Industrial Research.	Dehydrated Meat.	30/IV
Desmangeot, L.	Sublimation and Pseudo-Sublimation.	8/IV
Dewey, D. H.	Evaporative Cooling of Fruits and Vegetables.	134/III
Digney, F. J., *et al.*	Design and Use of an Electronic Pressure Controller.	122/II
Doehaerd, Th., *et al.*	Direct Determination of the Sublimation Energy of Carbon.	33/I
Donovan. G. E., *et al.*	Colour in Electron Microscopy.	19/II
Dunning, K. L., *et al.*	A Portable 250-Kilovolt Accelerator.	54/IV
Durand, E., *et al.*	A Portable 250-Kilovolt Accelerator.	54/IV
Durant, J. H.	The Thoughts and Actions Leading to the Formation of the C.V.T.	132/I
Duesing, W.	High Vacuum Glass-Kovar Melts.	60/IV
Dyke, W. P., *et al.*	Field Emission: Large Current Densities, Space Charge, and the Vacuum Arc.	56/IV
Eastman Kodak Co., *et al.*	Vacuum Pumping Apparatus.	88/II
Eborall, R. J. L., *et al.*	Two Laboratory Furnaces for Melting Titanium Alloys.	41/III
Economos, G.	Behaviour of Refractory Oxides in Contact with Metals at High Temperatures.	67/II
Ehrhardt, S. A., *et al.*	Infra-Red Spectra of Evaporated Films	114/I
Electrical and Musical Industries, Ltd. *et al.*	Electron Discharge Devices.	29/III
Ellson, C. H., *et al.*	A Portable 250-Kilovolt Accelerator.	54/IV
Engel, A. Von, *et al.*	A New Electron Multiplication Process.	90/I
Epstein, L. I.	The Design of Optical Filters.	23/I
Eshaya, A. M., *et al.*	Rectification at Reduced Pressures.	44/II
Evans, D. M., *et al.*	Crystal Growth and Orientation in Deposits Condensed from the Vapour.	31/II
Evans, J. W., *et al.*	The Solubility of Oxygen in Gamma Iron.	125/I
Farmer, E. C., *et al.*	Preparation of a Tritiated Self-Luminescent Material.	112/I
Fert, C., *et al.*	Aluminium Films in Electron Microscopy : The Production of Replicas with a Great Resolving Power.	105/II
Fields, M., *et al.*	Preparation of a Tritiated Self-Luminescent Material.	112/I
Figour, H., *et al.*	The Assessment of the Quality of Zirconium Powder Intended for Use in the Production of Vacuum Tubes.	76/I
Fish, F. H., *et al.*	Spectroscopic Study of Small Samples in a Hollow-Cathode Discharge.	47/I
Fitz Osborne, F. J.	A Simplified Ionisation Gauge Circuit.	52/II
Florescu, N. A.	A Compression Vacuum Gauge with a Large Working Range.	11/II
Flosdorf, E. W., *et al.*	Process for the Desiccation of Labile Aqueous Materials.	12/IV
Forman, R.	A Vacuum Valve to Provide Small Controlled Leak Rates.	137/I
Forrest, J. S.	Methods of Increasing the Electrical Conductivity of Surfaces.	91/I
Fox, G. L.	A Large-Diameter Reciprocating-Action Vacuum Valve.	120/II
Friedrich, R. J., *et al.*	Analysis of Synthesis Gas. Rapid and Highly Accurate Method.	21/IV
Frisch, N. W., *et al.*	Rectification at Reduced Pressures.	44/III
Fujiki, Y., *et al.*	On the Structure Loosening of Metal Films Deposited from the Vapour.	103/III
Garbe, H. W.	HF Brazes High Vacuum Joints.	157/I
Garden, N., *et al.*	Special Equipment for Carbon^{14} Work	91/III
Garfitt, R. G., *et al.*	An Electron Diffraction Instrument.	16/II
Garton, W. R. S.	A Demountable Flash-Discharge Tube for Production of the Lyman Continuum.	154/I
Geach, G. A., *et al.*	Arc-Melting of Non-Metallic Materials and a Re-determination of the Beryllia-Thoria System.	109/II
Gerds, A. F., *et al.*	Determination of Hydrogen in Magnesium, Lithium and Magnesium-Lithium Alloys.	85/III
Gibbs, D. S., *et al.*	A Recording Mercurial Manometer for the Pressure Range 0-760 mm. of Mercury.	47/II
Gibbs-Smith, A. J.	High Vacuum Melting. Recent Continental Developments.	39/III
Gilbert, H., *et al.*	Forging of Arc Melted Chromium.	84/III
Gillam, E., *et al.*	A Powder Camera Modification for High Temperature Photographs.	64/I
Gleiser, M., *et al.*	The Solubility of Oxygen in Gamma Iron.	125/I
Glick, D., *et al.*	Simple and Efficient Freezing Drying Apparatus for the Preparation of Embedded Tissue.	28/IV
Glockler, G., *et al.*	The Pirani Gauge.	12/II
Goalwin, D. S., *et al.*	A Diaphragm-Type Capacitance-Type Micromanometer for Very Low Differential Pressures.	49/II
Gobrecht,H., *et al.*	Radiation from Freshly Evaporated Layers of Metals and Salts.	48/I

Vol. 3 1953

Cumulative Author Index, Vol. III (1953)

Contd.

(For Abstract Nos. 1/I-68/I; 1/II-40/II; 1/III-52/III; 1/IV-18/IV see January issue)
(For Abstract Nos. 69/I-131/I; 41/II-84/II; 53/III-96/III; 19/IV-37/IV see April issue)

Author	Title of Publication	Abstr. No.
Godley, P., *et al.*	Deposition of Metal on a Non-metallic Support.	16/III
Gold, N., *et al.*	Apparatus for Vacuum Firing of Porcelain Dental Articles.	186/I
Goldfinger, P., *et al.*	Direct Determination of the Sublimation Energy of Carbon.	33/I
Goldsbarry, A. W., *et al.*	Falling-Film Type of Vacuum Still with Rotating Partial Condenser.	87/III
Gollan, F., *et al.*	A Large Capacity, All-Glass Dispersion Oxygenator and Pump.	41/I
Gomer, R.	A Novel Method for the Estimation of Very Low Pressures.	9/II
Goodner, K., *et al.*	Studies on the Stability of Lyophilised BCG Vaccine.	29/IV
Gray, R. V.	A Versatile Vacuum Melting Furnace.	124/III
Green, T. D., *et al.*	Preparation of Uniformly Dispersed Specimens of Particulate Matter for Electron Microscopy.	54/II
Greenland, K. M., *et al.*	Interference Filters in Optics.	25/I
Greve, L. de, *et al.*	Thin Glass Layers as Supports for Electron Microscopy.	24/II
Griffith, C. B., *et al.*	Determination of Hydrogen in Magnesium, Lithium and Magnesium-Lithium Alloys.	85/III
Grisard, J. W., *et al.*	Metal Ebulliometers for Vapour Pressure Measurement.	84/II
Grube, W. L., *et al.*	Pre-shadowed Replicas for Electron Metallography.	17/II
Grunberg, L., *et al.*	Kramer and Russell Effects with Single Crystals of Zinc.	179/I
Hacking, K., *et al.*	The Preparation of Titanium Dioxide Beam-Splitters of Large Surface Area.	106/III
Haggerty, A., *et al.*	Analysis of Synthesis Gas. Rapid and Highly Accurate Method.	21/IV
Haine, M. E., *et al.*	The Electronic Clean-Up of Gases in Sealed-Off Vacuum Systems.	134/I
Hall, G. L., *et al.*	Positive and Negative Ions in Cathode-Ray Tubes.	153/I
Haltmeier, A., *et al.*	Vacuum Dryer for Drying of Temperature-Sensitive Materials Supplied in Paste Form.	34/III
Hammer, B. W., *et al.*	Process for Producing Milk Fat Concentrate and Butter.	135/III
Hammond Engineering Co. Ltd., *et al.*	Improvements i.o.r.t. Pumps having Sliding Vanes.	5/II
Harper, M. E., *et al.*	Arc-Melting of Non-Metallic Materials and a Re-determination of the Beryllia-Thoria System.	109/II
Harries, W. L.	The Electrical Breakdown of Air between Insulators.	32/IV
Harries, W. L., *et al.*	A New Electron Multiplication Process.	90/I
Harrison, E. R.	Glass Leak and Control Valves.	136/I
Hartman, R. E., *et al.*	Preparation of Uniformly Dispersed Specimens of Particulate Matter for Electron Microscopy.	54/II
Harwood, J. H.	Industrial Applications of Activated Alumina.	64/II
Hass, G., *et al.*	Mirror Coatings for Low Visible and High Infra-Red Reflectance.	103/I
Heal, H. T., *et al.*	A Powder Camera Modification for High Temperature Photographs.	64/I
Heard, H. G., *et al.*	Transfer of Anode Metal in D.C. Non-Sparking Discharges in High Vacuum.	15/IV
Heavens, O. S.	The Design and Use of the Electron Diffraction Camera.	15/II
Heavens, O. S.	The Contamination in Evaporated Films by the Materials of the Source.	7/III
Helwig, G.	Electrical Conductivity and Structure of Sputtered Cadmium Oxide Layers.	70/III
Hess, G. E., *et al.*	Preparation of Uniformly Dispersed Specimens of Particulate Matter for Electron Microscopy.	54/II
Heuser, S. G., *et al.*	Behaviour of Fumigants During Vacuum Fumigation. I. Penetration of Methyl Bromide into Boxes of Dates.	73/III
Heyn, F. A., *et al.*	The Synchrocyclotron at Amsterdam. IV. Details of Construction and Ancillary Equipment.	37/IV
Hinsch, J., *et al.*	Improvements i.o.r.t. Two Stage Liquid-Ring Air-Pumps.	92/II
Hisatake, K., *et al.*	A Study on Diffusion Pump Oil by the Mass Spectrometer and the Gas Analysis of the Final Vacuum.	90/II
Holland, L.	The Effect of Vapour Incidence on the Structure of Evaporated Aluminium Films.	160/I
Holland, L., *et al.*	Multifilament Vapour Source and Spherical Holder for Interference Filters.	9/III
Holland, L., *et al.*	The Preparation of Titanium Dioxide Beam-Splitters of Large Surface Area.	106/III
Holland, L., *et al.*	The Coating of Plastics by Vacuum Evaporation for Functional and Scientific Purposes.	115/III
Holmes, J. M., *et al.*	Adsorption by Evaporated Copper Films at 78°K. Part I. Krypton and Hydrogen.	40/IV
Holmes, J. M., *et al.*	Adsorption by Evaporated Copper Films at 78°K. Part II. Krypton and Oxygen.	41/IV
Hooven, F., *et al.*	A Large Capacity, All-Glass Dispersion Oxygenator and Pump.	41/I
Horneman, H. C., *et al.*	Process for Producing Milk Fat Concentrate and Butter.	135/III
Horst, H. V., *et al.*	The Pirani Gauge.	12/II
Horton, W. S., *et al.*	Determination of Oxygen in Chromium by the Vacuum Fusion Method.	128/III
Hosler, W. R., *et al.*	Titanium Dioxide Rectifiers.	15/I

Vol. 3 195

Cumulative Author Index, Vol. III (1953)

Contd.

(For Abstract Nos. 1/I-68/I; 1/II-40/II; 1/III-52/III; 1/IV-18/IV see January issue)
(For Abstract Nos. 69/I-131/I; 41/II-84/II; 53/III-96/III; 19/IV-37/IV see April issue)

Author	Title of Publication	Abstr. No.
Howerton, K. H., *et al.*	A Portable 250-Kilovolt Accelerator.	54/IV
Hussong, R. V., *et al.*	Process for Producing Milk Fat Concentrate and Butter.	135/III
Inn, E. C. Y., *et al.*	Intensity Measurements in the Vacuum Ultra-Violet.	99/I
Itterbeek, A. van, *et al.*	Thin Glass Layers as Supports for Electron Microscopy.	24/II
Jacobs, J. A., *et al.*	Increased Sensitivity of Leak Detection with Hydrogen.	123/II
Jaeckel, R., *et al.*	Vacuum Pumps for Pressures below One Torr.	6/I
James, D. H., *et al.*	A Simple Gas-Flow-Control of High Efficiency.	9/I
Johansen, H. A., *et al.*	Forging of Arc Melted Chromium.	84/III
Jones, D. A.	Mercury Vapour as a Health Hazard.	115/II
Jones, G. O., *et al.*	A Miniature Helium Liquefier-Cryostat of Cascade Type.	125/II
Kaechele, W. H., *et al.*	A Diaphragm-Type Capacitance-Type Micromanometer for Very Low Differential Pressures.	49/II
Kaesberg, P., *et al.*	The Breaking of Tobacco Mosaic Virus.	52/IV
Keith, W. P.	Molybdenum-Wound Hydrogen Atmosphere Furnace.	55/I
Kelly, E. J.	Alkali-Refined Linseed Oil.	116/I
Kemball, C., *et al.*	Catalysis on Evaporated Metal Films. 1. The Efficiency of Different Metals for the Reaction between Ammonia and Deuterium.	49/I
Kermer, M. J., *et al.*	De-airating and De-oiling Citrus Juices.	93/III
Kestner Evaporator and Engineering Co. Ltd., *et al.*	Improvements i.o.r.t. the Concentration by Evaporation of Heat Sensitive Liquors.	119/III
Keywell, F.	A Mechanism for Sputtering in the High Vacuum based upon the Theory of Neutron Cooling.	28/III
Kihara, T.	Mathematical Theory of Electrical Discharges in Gases.	11/I
Kington, G. L., *et al.*	Adsorption by Evaporated Copper Films at 78°K. Part I. Krypton and Hydrogen.	40/IV
Kington, G. L., *et al.*	Adsorption by Evaporated Copper Films at 78°K. Part II. Krypton and Oxygen.	41/IV
Kirshenbaum, A. D., *et al.*	Direct Determination of Oxygen in Rubber.	176/I
Kistemaker, J.	Progress in High Vacuum Technique.	70/I
Kitchener, J. A., *et al.*	The Solubility of Oxygen in Gamma Iron.	125/I
Klemenc, A., *et al.*	Heat Conductivity of Gases at Low Pressures.	4/IV
Klumb, H., *et al.*	Supersonic Speeds in High Vacuum.	91/II
Kober, H.	The Optical Constants of Gold and Silver in the Ultra-Violet.	166/I
Koenig, H.	The Formation of Adsorbed Layers in Vacuum Chambers and their Detection by Electrons.	77/I
Koppen, G. M. van.	A Metal Vacuum Valve.	76/II
Korbelak, A., *et al.*	Plating as an Aid in the Brazing of Stainless Steel.	95/I
Kramers, W. J., *et al.*	A High Temperature Resistance-Heated Vacuum Furnace.	123/III
Kurz, G.	A New Apparatus for the Determination of Moisture Content.	171/I
Lafourcade, L.	Study of the Crystalline Grain in Thin Films of Gold or Palladium or Gold-Palladium Alloys Prepared by the Thermal Evaporation Technique.	161/I
Lauer, E. J., *et al.*	Transfer of Anode Metal in D.C. Non-Sparking Discharges in High Vacuum.	15/IV
Lawrance, R. B., *et al.*	Improvements i.o.r.t. High Vacuum Oil Diffusion Pumps.	42/II
Lawrance, R. B., *et al.*	High Vacuum Device.	89/II
Lawrance, R. B., *et al.*	Improvements i.o.r.t. High Vacuum Diffusion Pumps.	86/II
Lawrence, P. H., *et al.*	Improvements i.o.r.t. the Production of Coatings on Ferrous Articles.	123/I
Leader, D.	Electrical Breakdown in Vacuum.	31/IV
Leger, P., *et al.*	Uniform Surface Metallisation by Evaporation from One or More Emission Surfaces.	56/III
Lehovec, K., *et al.*	Evaporation of Germanium Films from a Carbon Crucible.	100/III
Levi, R.	New Dispenser Type Thermionic Cathode.	36/IV
Leviant, I.	Introduction to the Vacuum Concrete Process	48/III
Lewin, J.	Application of Vacuum Technique to the Production of Blood Plasma and Analogous Biological Products.	49/IV
Libbey-Owens-Ford Glass Company	Light-Transmissive Electrically Conducting Optical Articles Suitable for Use as a Lens, a Window or Windshield, or the Like.	13/III
Lind, E. R., *et al.*	Development of a Large, Linear Jet, Mercury Diffusion Pump Having High Pumping Speeds in the 10^{-6} mm. Mercury Absolute Pressure Range.	41/II
Linder, E. G., *et al.*	The Use of Radioactive Materials for the Generation of High Voltage.	14/I
Llewellyn Jones, F.	Electrical Discharges.	89/I
Llewellyn Jones, F.	Electrical Discharges and the Vacuum Physicist.	152/I
Lowe, D. S., *et al.*	Infra-Red Molecular Emission Excited in a Glow Discharge.	2/IV

Cumulative Author Index, Vol. III (1953)

Contd.

(For Abstract Nos. 1/I-68/I; 1/II-40/II; 1/III-52/III; 1/IV-18/IV see January issue)
(For Abstract Nos. 69/I-131/I; 41/II-84/II; 53/III-96/III; 19/IV-37/IV see April issue)

Author	Title of Publication	Abstr. No.
Lyophile Cryochem Corpn., *et al.*	Process for the Desiccation of Labile Aqueous Materials.	12/IV
Macdonald, A., *et al.*	Evaporation of Germanium Films from a Carbon Crucible.	100/III
Madeley, R. S. B., *et al.*	Improvements i.o.r.t. Vacuum Insulated Systems for Conveying or Storing Liquefied Gases.	126/II
Malé, D.	On the Choice of Measurements to be Made for Simultaneous Determination of the Optical Constants and Thickness of Thin Absorbing Films.	109/III
Mallett, W. *et al.*	Determination of Hydrogen in Magnesium, Lithium and Magnesium-Lithium Alloys.	85/III
Malmberg, P. R., *et al.*	Increased Sensitivity of Leak Detection with Hydrogen.	123/II
Malmstrom, B. G., *et al.*	Simple and Efficient Freezing Drying Apparatus for the Preparation of Embedded Tissue.	28/IV
Matsuda, K., *et al.*	A Study on Diffusion Pump Oil by the Mass Spectrometer and the Gas Analysis of the Final Vacuum.	90/II
McCulloch, R. J., *et al.*	The Design of High Vacuum Systems. Part I & II.	71/I
Meinken, R. H., *et al.*	A Technique for Making Molybdenum Crucibles for Ceramic Research.	129/I
Methfessel, S.	Metal Evaporation in High Vacuum.	98/III
Metropolitan Vickers Electrical Co. Ltd.	Improvements in Apparatus for Introducing Specimens into an Evacuated Chamber.	58/II
Middleton, J. L., *et al.*	Improvements i.o.r.t. High Vacuum Diffusion Pumps.	86/II
Mielenz, K. D., *et al.*	On the Theory of the Quartz Fibre Manometer.	94/II
Miller, M. A., *et al.*	Vacuum Tightness of Welded and Brazed Aluminium Containers.	94/I
Miller, R., *et al.*	Studies on the Stability of Lyophilised BCG Vaccine.	29/IV
Miller, W. W., *et al.*	Carbon Dioxide.	111/II
Mitchell, J. T., *et al.*	Coupling Glass Tubing to Other Materials.	73/II
Mojonnier Bros., Co.	Improvements i.o.r.t. a Process and Apparatus for Evaporating Liquids.	77/III
Moore, J. H., *et al.*	Vacuum Casting Apparatus.	125/III
Morrison, J.	A Controlled Gas Leak.	82/I
Mueller, E. W.	Field Emission Microscopy.	53/II
Muramatsu, K., *et al.*	A Large Unwelded Vacuum Gate Valve.	119/II
Myers, H. P.	A Simple Varying Capacitor Method for the Measurement of Contact Potential Difference in High Vacuum.	57/IV
Nakamura, M., *et al.*	On the PbS Photoconductive Cell.	163/I
National Research Corp.	Improvements i.o.r.t. High Vacuum Oil Diffusion Pumps.	42/II
National Research Corp., *et al.*	Deposition of Metal on a Non-metallic Support.	16/III
National Research Corp.	Apparatus and Process for Forming a Metal-Free Stripe on a Web of Metal-Coated Sheet Material.	17/III
National Research Corp.	Apparatus and Process for Coating a Substrate with a Metal.	59/III
National Research Corporation, *et al.*	Vacuum Casting Apparatus.	125/III
National Research Corporation, *et al.*	Improvements i.o.r.t. Stills.	130/III
National Research Corporation, *et al.*	Improvements i.o.r.t. High Vacuum Diffusion Pumps.	86/II
National Research Corporation, *et al.*	High Vacuum Device.	89/II
Nelson, R. G., *et al.*	Forging of Arc Melted Chromium.	84/III
Newbound, K. B., *et al.*	Spectroscopic Study of Small Samples in a Hollow-Cathode Discharge.	47/I
Newman, N. J., *et al.*	Multifilament Vapour Source and Spherical Holder for Interference Filters.	9/III
Niebuhr, J.	The Electrical Resistivity of Thin Tin Films with Lattice Defects.	20/III
Nier, A. O. C.	The Mass Spectrometer.	139/I
Noeller, H. G., *et al.*	Vacuum Pumps for Pressures below One Torr.	6/I
Normand, L. E., *et al.*	Evaluation of Vacuum Gasket Materials.	71/II
North American Aviation Inc.	A Vapour Pressure Chart for Metals.	52/I
Norton, F. J.	Helium Diffusion Through Glass.	43/IV
Nu-Dent Porcelain Studios Inc., *et al.*	Apparatus for Vacuum Firing of Porcelain Dental Articles.	186/I
Numerof, P., *et al.*	Simple Laboratory Evaporator.	78/III
Okress, E. C., *et al.*	Plating as an Aid in the Brazing of Stainless Steel.	95/I
Oliver, G. D., *et al.*	Metal Ebulliometers for Vapour Pressure Measurement.	84/II
Olney, M. J.	Thermal Etching of Iron and Steel.	59/I
Opler, A., *et al.*	Device for Preparing Gas Mixtures for Calibration.	39/IV
Pack D. C.	The Motion of a Gas Cloud Expanding into a Vacuum.	98/I

Vol 3 195

Cumulative Author Index, Vol. III (1953)

Contd.

(For Abstract Nos. 1/I-68/I; 1/II-40/II; 1/III-52/III; 1/IV-18/IV see January issue)
(For Abstract Nos. 69/I-131/I; 41/II-84/II; 53/III-96/III; 19/IV-37/IV see April issue)

Author	Title of Publication	Abstr. No.
Paemel, O. van	Thermal Conductivity of Glass Wool at Low Temperatures under Low Pressure of the Air.	107/I
Page, R. S., *et al.*	Improvements in Apparatus for Introducing Specimens into an Evacuated Chamber.	58/II
Page, R. S., *et al.*	An Electron Diffraction Instrument.	16/II
Palme, R.	Tungsten and Molybdenum in Vacuum Engineering.	53/I
Paneth, F. A.	The Micro-Analysis of the Inert Gases.	111/I
Parsegian, V. L., *et al.*	Apparatus for Supplying Charge Material to Mass Spectrometers.	60/II
Patrick, E. A. K.	Bursting Disc Assembly for Alternating Pressure and Vacuum at Elevated Temperatures.	96/I
Perls, T. A., *et al.*	A Diaphragm-Type Capacitance-Type Micromanometer for Very Low Differential Pressures.	49/II
Perrot, M., *et al.*	Contribution to the Study of the Conductivity of Silicon.	22/III
Perrot, M., *et al.*	The Influence of the Electrical Field on the Performance of Thin Films.	21/III
Philips' Gloeilampenfabrieken, N.V.	Improvements i.o.r.t. Metal Cans Sealed in an Airtight Manner.	96/III
Phillips, C. S. G., *et al.*	A Simple Gas-Flow-Control of High Efficiency.	9/I
Pilny, M. J.	A Small Pirani Gauge for Measurements of Non-Steady Low Pressures.	95/II
Pohlack, H.	On the Problem of Reflection Reduction in Optical Glass at Light Incident at an Oblique Angle.	26/I
		26/I
Pressey, D. C.	Temperature-Stable Capacitance Pressure Gauges.	48/II
Prugne, P., *et al.*	Uniform Surface Metallisation by Evaporation from One or More Emission Surfaces.	56/III
Pure Oil Company, The, *et al.*	Falling-Film Type of Vacuum Still with Rotating Partial Condenser.	87/III
Putner, T., *et al.*	The Preparation of Titanium Dioxide Beam-Splitters of Large Surface Area.	106/III
Quam, S. N., *et al.*	Process for Producing Milk Fat Concentrate and Butter.	135/III
Quurke, W. F. W.	Ultra Violet Photography.	28/I
Rainsforth, R., *et al.*	Improvements i.o.r.t. the Production of Coatings on Ferrous Articles.	123/I
Rathkamp, W. R., *et al.*	Evaluation of Vacuum Gasket Materials.	71/II
Reavell, J. A., *et al.*	Improvements i.o.r.t. the Concentration by Evaporation of Heat Sensitive Liquors.	119/III
Redhead, P. A.	A Stabilised Ion Gauge Control Unit.	14/II
Rees, J. A., *et al.*	Two Laboratory Furnaces for Melting Titanium Alloys.	41/III
Regel, F.	Electrical High Temperature Furnaces, Fitted with Molybdenum Resistance Heaters.	183/I
Reinhardt, K., *et al.*	Simple Laboratory Evaporator.	78/III
Reinhart, F.	Glare Reducing Glass for Goggles.	27/I
Reinhart, F.	The Bonding of Siliceous and Metallic Components.	59/IV
Renzema, T. S.	Electron Diffraction Investigation of Polished Surfaces.	121/I
Reynolds, F. W., *et al.*	Mean Free Paths of Electrons in Evaporated Metal Films.	64/III
Rice, R. V., *et al.*	The Breaking of Tobacco Mosaic Virus Using a New Freeze Drying Method.	52/IV
Riddiford, L.	The Cooling and Baffling of Diffusion Pumps.	43/II
Ring, J., *et al.*	Multilayer Dielectric Reflecting Films at Wavelengths between 4,000 and 4,600 Å.	164/I
Robens, E., *et al.*	Supersonic Speeds in High Vacuum.	91/II
Roberts, N. W., *et al.*	Improvements i.o.r.t. Vacuum Insulated Systems for Conveying or Storing Liquefied Gases.	126/II
Robin, S.	Measurement of the Reflective Power of Thick Metallic Layers (Au, Pt, Cr) in the Schumann Range. Determination of Optical Constants.	100/I
Robin, S., *et. al.*	Apparatus for the Measurement of Reflecting Power in the Vacuum Ultra-Violet.	101/I
Rosen, J., *et al.*	Evaporation of Germanium Films from a Carbon Crucible.	100/III
Rouze, S. R., *et al.*	Pre-Shadowed Replicas for Electron Metallography.	17/II
Rowland, P. B.	Preparation of Specimens for the Electron Diffraction Camera.	103/II
Rubury, J. M., *et al.*	Improvements i.o.r.t. Pumps Having Sliding Vanes.	5/II
Russell, A. S., *et al.*	Vacuum Tightness of Welded and Brazed Aluminium Containers.	94/I
Russell, L. N., *et al.*	On the Preparation of Magnesium Targets from MgO.	47/III
Sassa, N., *et al.*	On the PbS Photoconductive Cell.	163/I
Sawyer, G. A.	Unbacked Thin Films.	17/IV
Scanlon, W. W., *et al.*	Infra-Red Molecular Emission Excited in a Glow Discharge.	2/IV
Schmid, E.	Casting Under Extreme Conditions.	80/III
Scholz, O., *et al.*	Supersonic Speeds in High Vacuum.	91/II
Schoenheit, E., *et al.*	On the Theory of the Quartz Fibre Manometer.	94/II
Schroeder, H. H., *et al.*	Mirror Coatings for Low Visible and High Infra-Red Reflectance.	103/I

Vol. 3 1953

Cumulative Author Index, Vol. III (1953)

Contd.

(For Abstract Nos. 1/I-68/I; 1/II-40/II; 1/III-52/III; 1/IV-18/IV see January issue)
(For Abstract Nos. 69/I-131/I; 41/II-84/II; 53/III-96/III; 19/IV-37/IV see April issue)

Author	Title of Publication	Abstr. No.
Schroers, F. H., *et al.*	Paper for Punched Cards.	147/I
Schroers Jacquard Papier Industrie, *et al.*	Paper for Punched Cards.	147/I
Schunck, H.	Dust Filters for Rotary Vacuum Pumps.	6/II
Schwarz, H.	Method and Apparatus for the Measurement of Very Small Gas Pressures.	8/I
Schwarz, H.	Method of Realising Very High Vacua by Ionisation. Design of an Electronic Pump.	1/II
Schwarz, H.	Methods of Obtaining High Vacuum by Ionisation. Construction of an ' Electronic Pump '.	85/II
Seger, H.	The Moisture Content of Dried Plasma and its Determination.	10/IV
Seiter, J. G.	Vacuum Metallising in the Plastics Industry.	3/III
Senseney, C. A., *et al.*	Preparation of Uniformly Dispersed Specimens of Particulate Matter for Electron Microscopy.	54/II
Shackleton, L. R. B., *et al.*	Improvements i.o.r.t. Stills.	130/III
Siemen, O., *et al.*	Improvements i.o.r.t. Two Stage Liquid-Ring Air-Pumps.	92/II
Silberg, P. A., *et al.*	Positive and Negative Ions in Cathode-Ray Tubes.	153/I
Sites, J. R., *et al.*	Mass Spectrometer Studies of High Vacuum Materials.	28/II
Slavin, W., *et al.*	A Portable 250-Kilovolt Accelerator.	54/IV
Smith, E. S., *et al.*	Device for Preparing Gas Mixtures for Calibration.	39/IV
Smyth, H. T., *et al.*	A Technique for Making Molybdenum Crucibles for Ceramic Research.	129/I
Snaddon, R., *et al.*	Safety Trap for Vacuum Line.	80/II
Sommer, A., *et al.*	Electron Discharge Devices.	29/III
Sotavalta, O.	Flight-Tone and Wing-Stroke Frequency of Insects and the Dynamics of Insect Flight.	68/I
Speed, F. L.	A Design of Pressure/Vacuum Valve.	121/II
Spragg, W. T., *et al.*	A Single Air Ejector as a Suction Motor for Dry Boxes.	45/II
Stafford, E., *et al.*	Colour in Electron Microscopy.	19/II
Stahmann, M. A., *et al.*	The Breaking of Tobacco Mosaic Virus Using a New Freeze Drying Method.	52/IV
Steinhaus, J. F., *et al.*	Development of a Large, Linear Jet, Mercury Diffusion Pump Having High Pumping Speeds in the 10^{-6} mm. Mercury Absolute Pressure Range.	41/II
Stevens, A. H., *et al.*	Improvements i.o.r.t. a Process and Apparatus for Evaporating Liquids.	77/III
Stevens, C. M.	A Vacuum Lock for Routine Solid Analyses with a Mass Spectrometer.	59/II
Stevens, C. M., *et al.*	Apparatus for Supplying Charge Material to Mass Spectrometers.	60/II
Stilwell, G. R., *et al.*	Mean Free Path of Electrons in Evaporated Metal Films.	64/III
Strashun, S. I., *et al.*	Puffed Powder from Juice.	76/III
Streng, A. G., *et al.*	Direct Determination of Oxygen in Rubber.	176/I
Suganuma, R., *et al.*	On the Structure Loosening of Metal Films Deposited from the Vapour.	103/III
Sutherland, N., *et al.*	The Coating of Plastics by Vacuum Evaporation for Functional and Scientific Purposes.	115/III
Svec, H. J., *et al.*	A Recording Mercurial Manometer for the Pressure Range 0-760 mm. of Mercury.	47/II
Tait, T. R., *et al.*	Coupling Glass Tubing to Other Materials.	73/II
Talburt, W. F., *et al.*	Puffed Powder from Juice.	76/III
Talley, R. M., *et al.*	Infra-Red Molecular Emission Excited in a Glow Discharge.	2/IV
Tallman, J. C.	Ejectors Show Low First Cost.	44/II
Taylor, W. E., *et al.*	On the Preparation of Magnesium Targets from MgO.	47/III
Teed, P. L.	Titanium : A Survey.	81/III
Telefunk, G. F. Drahtlose Telegraphie, m.b.H.	Method for the Deposition of a Thin Metal Layer on the Rough Surface of the Screen of a Cathode Ray Tube.	62/III
Thomas, E.	Calculation of the Rate of Diffusion of Hydrogen Through a Plate of Palladium.	44/IV
Thompson, J. E.	Distillation Apparatus for Refining Fats and Oils.	88/III
Thorne, A. L. C.	Recovery of Caprinised and Lapinised Rinderpest Viruses from Condensed Water Vapour Removed During Desiccation.	53/IV
Titangesellschaft m.b.H.	Improvements i.o.r.t. the Manufacture of, and Coating by Titanium or Alloys of Titanium.	82/III
Tolansky, S.	Shadow Microscopy for Measurement of Height.	105/I
Tolbert, B. M., *et al.*	Special Equipment for Carbon^{14} Work.	91/III
Tombs, N. C., *et al.*	Thermodynamic Properties of Silicon Monoxide. An Experimental Study of the Equilibria $SiO_2 + Si \rightleftharpoons 2SiO$ and $SiO_2 + H_2 \rightleftharpoons SiO + H_2O$.	51/I
Tortosa, J., *et al.*	Contribution to the Study of the Conductivity of Silicon.	22/III
Townsend, J. W., Jr.	Radiofrequency Mass Spectrometer for Upper Air Research.	20/II
Trolan, J. K., *et al.*	Field Emission : Large Current Densities, Space Charge, and the Vacuum Arc.	56/IV
Tufts, C. F.	Determination of Surface Geometry and Structure by Microscopy and Diffraction.	36/I

Cumulative Author Index, Vol. III (1953)

Contd.

(For Abstract Nos. 1/I-68/I; 1/II-40/II; 1/III-52/III; 1/IV-18/IV see January issue)
(For Abstract Nos. 69/I-131/I; 41/II-84/II; 53/III-96/III; 19/IV-37/IV see April issue)

Author	Title of Publication	Abstr. No.
Turkevich, J., *et al.*	Carbon Dioxide.	111/II
Turnbull, J. C.	A High Conductivity Glass-to-Metal Seal.	18/IV
Turner, A. F., *et al.*	Mirror Coatings for Low Visible and High Infra-Red Reflectance.	103/I
Tuynman, C. A. F., *et al.*	Thin Glass Layers as Supports for Electron Microscopy.	24/II
Tyler, J. E., *et al.*	Infra-Red Spectra of Evaporated Films.	114/I
U.S. Atomic Energy Commission.	Diffusion Measuring Meter.	24/II
U.S. Atomic Energy Commission.	Apparatus for Supplying Charge Material to Mass Spectrometers.	60/II
U.S. Atomic Energy Commission, *et al.*	Carbon Dioxide.	111/II
Vanderveer, W. R., *et al.*	Brazing Vacuum-Tight Joints in High Nickel Alloys.	33/II
Veelen, G. F. van, *et al.*	Thin Glass Layers as Supports for Electron Microscopy.	24/II
Vodar, B., *et al.*	Apparatus for the Measurement of Reflecting Power in the Vacuum Ultra-Violet.	101/I
Vogt, A.	Valve Arrangement for Vacuum Pump Units.	36/II
Vogt, A.	Improvements i.o.r.t. Devices for Melting and Casting Materials under Air-Exclusion.	126/III
Vogt, A.	Improvements i.o.r.t. Condensers for Oil Diffusion or Vapour Jet Vacuum Pumps.	87/II
Vyner, E.	Aspects of the Evaporation and Drying Process in the Glue and Gelatine Industry.	33/III
Waelbroeck, F.	On the Mechanism of Evaporation of Graphite.	34/I
Waelbroeck, F., *et al.*	Direct Determination of the Sublimation Energy of Carbon.	33/I
Wagener, S.	The Use of Getters for the Production of Very High Vacua.	75/I
Wagener, S.	Sorption of Gases at Very Low Pressures by Thorium Powder.	135/I
Wal, R. J. van der, *et al.*	The Semi-Continuous Deodorisation of Oils in All-Glass Equipment.	51/III
Wallace, R. A., *et al.*	Brazing Vacuum-Tight Joints in High Nickel Alloys.	33/II
Walton, J. R.	High Temperature Mass Spectrometer Oven.	101/II
Warren, H. G., *et al.*	Vacuum Pumping Apparatus.	88/II
Warring, R. H.	Product Finishing by Metallic Deposition in Vacuo.	1/III
Watanabe, K.	Improvements of Grating Efficiency in the Vacuum Ultra-Violet by Platinising.	167/I
Watanabe, K., *et al.*	Intensity Measurements in the Vacuum Ultra-Violet.	99/I
Watson, G. G., *et al.*	Heat Transfer to Mercury.	30/II
Webb, C. G., *et al.*	A Single Air Ejector as a Suction Motor for Dry Boxes.	45/II
Wegmann, L.	Specimen Changes and Electron Optical System in Electron Microscopes.	57/II
Weissler, G. L., *et al.*	Work Functions of Gas-Coated Tungsten and Silver Surfaces.	58/IV
Welch, A. J. E., *et al.*	Thermodynamic Properties of Silicon Monoxide, An Experimental Study of the Equilibria $SiO_2 + Si \rightleftharpoons 2SiO$ and $SiO_2 + H_2 \rightleftharpoons SiO + H_2O$.	
Westfall, F. O., *et al.*	Electrodeless Metal-Halide Lamps.	155/I
Wilcock, W. L., *et al.*	Multilayer Dielectric Reflecting Films at Wavelengths between 4,000 and 4,600 Å.	164/I
Williams, R. C.	A Method of Freeze Drying for Electron Microscopy.	55/II
Wilman, H., *et al.*	Crystal Growth and Orientation in Deposits Condensed from the Vapour.	31/I
Wilson, J. C., *et al.*	Improvements i.o.r.t. Stills.	130/III
Wilson, T. N., *et al.*	Work Functions of Gas-Coated Tungsten and Silver Surfaces.	58/IV
Wirth, H., *et al.*	Heat Conductivity of Gases at Low Pressures.	4/IV
Woods, J., *et al.*	Secondary Electron Emission from Barium Oxide.	34/IV
Woods, J., *et al.*	Changes in Secondary and Thermionic Emission from Barium Oxide during Electron Bombardment.	35/IV
Wright, D. A., *et al.*	Secondary Electron Emission from Barium Oxide.	34/IV
Wright, D. A., *et al.*	Changes in Secondary and Thermionic Emission from Barium Oxide during Electron Bombardment.	35/IV
Wright, K. H. R., *et al.*	Kramer and Russell Effects with Single Crystals of Zinc.	179/I
Wright, L. J.	The Histological Preparation of Human and Animal Tissues by Freeze-Drying.	50/IV
Yamaguchi, S.	A Note on ' Metallic Shadow-Casting Using a Nozzle System '.	101/III
Yerazunis, S., *et al.*	Design and Use of an Electronic Pressure Controller.	122/II
Young, R. S., *et al.*	Safety Trap for Vacuum Line.	80/II
Zemany, P. D.	Thermal Degradation of Polystyrene.	115/I
Zorll, U.	The Limit of Accuracy in the Measurement of the Thickness of a Thin Film Using the Koester Interference Double Prism.	104/I
Zwiebel, N., *et al.*	Carbon Dioxide.	111/II

Vol. 3 1953

Abbreviations and Publishers of Journals Quoted in this Volume

Acta Cryst.	Acta Crystallographica, Ejnar Munksgaard, Copenhagen, Denmark.
Acta Metallurg.	Acta Metallurgica, 57, East 55th Street, New York 22, N.Y., U.S.A.
A.E.C. Reports	U.S. Atomic Energy Commission Reports,
A.E.C.D. Reports	Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
A.E.R.E. Reports	U.K. Atomic Energy Research Establishment Reports, Her Majesty's Stationery Office, P.O. Box 569, London S.E.1, England.
Amer. Ceramic Soc. Bull.	The American Ceramic Society Bulletin, 2525, North High Street, Columbus 2, Ohio, U.S.A.
Amer. Export Industr.	American Exporter Industrial, Johnston Export Publishing Co., 386, Fourth Avenue, New York 16, N.Y., U.S.A.
Ann. Phys.	Annalen der Physik, Johann & Ambrosius Barth, Salomonstr. 18b, Leipzig C.1, Germany.
Analyt. Chem.	Analytical Chemistry, American Chemical Society, 1155, Sixteenth Street, N.W., Washington 6, D.C., U.S.A.
Appl. Sci. Res.	Applied Scientific Research, Martinus Nijhoff, The Hague, Holland.
Arch. Tech. Messen.	Archiv für Technisches Messen, R. Oldenbourg Verlag, Lotzbeckstrasse 2b, München, Germany.
Atomics	Leonard Hill Ltd., Stratford House, Eden Street, London N.W.1, England.
Aus Forschung und Produktion	Degussa, Deutche Gold- und Silber Scheideanstalt, Frankfurt/Main, Germany
Bild & Ton	Henschel Verlag Kunst und Gesellschaft, Oranienburger Strasse 67, Berlin N.4, Germany.
Biochim. Biophys. Acta	Biochimica et Biophysica Acta, Elsevier Publishing Co., Amsterdam, Holland.
Brit. Gel. & Glue R.A.	British Gelatine and Glue Research Association, 2a, Dalmeny Avenue, Holloway, London N.7, England.
Brit. J.Appl.Phys.	British Journal of Applied Physics, The Institute of Physics, 47, Belgrave Square, London S.W.1, England.
B.S.I.R.A. Bull.	British Scientific Instrument Research Association, Information Department, 'Sira', Southill, Elmstead Woods, Chislehurst, Kent, England.
Bull. Inst. Int. du Froid	Bulletin de l'Institut International du Froid, Institut International du Froid, 177, Boulevard Malesherbes, Paris 17e, France.
Canad. Chem. Process.	Canadian Chemical Processing, 137, Wellington Street West, Toronto, Ontario, Canada.
Canad. J. Phys.	Canadian Journal of Physics, The National Research Council, Ottawa, Canada.
Canad. J. Technol.	The Canadian Journal of Technology, The National Research Council, Ottawa, Canada.
Chemical Abstracts	The American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Chem. Engng.	Chemical Engineering, McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 36, N.Y., U.S.A.
Chem. Engng. Progr.	Chemical Engineering Progress, American Institute of Chemical Engineers, 120, East 41st Street, New York 17, N.Y., U.S.A.
Chem. Process Engng.	Chemical and Process Engineering, Leonard Hill Ltd. 17, Stratford Place. London W.1, England.
Chem. Ing. Tech.	Chemie Ingenieur Technik, Chemie Verlag, G.m.b.H., Hauptstrasse 129, Weinheim/Bergstrasse, Germany.
Compr. Air Mag.	Compressed Air Magazine, Compressed Air Magazine Co., Phillipsburg, N.Y., U.S.A.
Conference Opt. Soc. Amer.	Conference of the Optical Society of America, c/o Journal of the Optical Society of America, American Institute of Physics, 57, East 55th Street, New York 22, N.Y., U.S.A.
C.R. Acad. Sci., Paris	Comptes Rendus des Séances de L'Academie des Sciences, Messrs. Gauthier-Villar, 55, Quai des Grands-Augustine, Paris, France.
C.R. Comm. 1 & 2 I.I.F., Louvain	Comptes Rendus Commissions 1 & 2, Institut International du Froid, c/o Bulletin de l'Institut International du Froid, 177, Boulevard Malesherbes, Paris 17e, France.
Design & Res. Centre Bull.	Design and Research Centre Bulletin, Design and Research Centre, Goldsmiths' Hall, Foster Lane, London E.C.2, England.
Drug. & Cosm. Ind.	Drug and Cosmetic Industry, Drug Markets Inc., 101, West 31st Street, New York 1, N.Y., U.S.A.

Abbreviations and Publishers of Journals Quoted in this Volume

Contd.

D.S.I.R. Bibliography (& Reports)	Bibliography of Industrial Drying, Department of Scientific and Industrial Research, Her Majesty's Stationery Office, P.O. Box 569, London S.E.1, England.
Electronics	McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 18, N.Y., U.S.A.
Endeavour	Imperial Chemical Industries Ltd., 26, Dover Street, London W.1, England.
Engineer	The Engineer, Morgan Brothers (Publishers) Ltd., 28, Essex Street, Strand, London W.C.2, England.
Exp. Cell Res.	Experimental Cell Research, Academic Press Inc., 125, East 23rd Street, New York 10, N.Y., U.S.A.
Food Engng.	Food Engineering, McGraw-Hill Publishing Co. Inc. 330, West 42nd Street, New York 36, N.Y., U.S.A.
Giesserei	Giesserei Verlag G.m.b.H, Düsseldorf, Germany.
Glas-Email-Ker-Tech.	Glas-Email-Keramo-Technik, P.O. Box Verlag Brunke Garrels, Hamburg 26, Germany.
Glas & Hochvakuum Tech.	Glas und Hochvakuum Technik, Rudolf A. Lang Verlag, Leibnizstrasse 64, Berlin-Charlottenburg 4, Germany.
Glastech. Berichte	Glastechnische Berichte, Verlag der Deutschen Glastechnischen Gesellschaft, Untermainkai 12, Frankfurt (Main), Germany.
I.E.E. Paper	Institution of Electrical Engineers Paper (Special Print), c/o Proceedings of the Institution of Electrical Engineers, Institution of Electrical Engineers, Savoy Place, London W.C.2, England.
Industr. Chem.	Industrial Chemist, St. Margaret's Technical Press, Ltd., 35, Tothill Street, London S.W.1, England.
Industr. Engng. Chem.	Industrial and Engineering Chemistry American Chemical Society, 20th and Northampton Streets, Easton, Pa., U.S.A.
Industr. Finish.	Industrial Finishing, Arrow Press Ltd. 157, Hagden Lane, Watford Herts., England.
Instruments	Instrument Publishing Co. Inc., 921, Ridge Avenue, Pittsburgh 12, Pa., U.S.A.
Invest. Phys.	Investigationes Physicae, 40, Carlton Crescent, Summer Hill, Sydney, N.S.W., Australia.
Iron Age	Chilton Co. Inc., Chestnut and 56th Streets, Philadelphia 39, Pa., U.S.A.
Iron & Steel	The Louis Cassier Co., Ltd., Dorset House, Stamford Street, London S.E.1, England.
Jenaer Jahrbuch	Kommissionsverlag Gustav Fischer, Jena, Germany.
J. Amer. Ceramic Soc.	Journal of the American Ceramic Society, American Ceramic Society, 2525, N. High Street, Columbus 2, Ohio, U.S.A.
J. Amer. Oil Chem. Soc.	Journal of the American Oil Chemists Society, The American Oil Chemists' Society, 35, E. Wacker Drive, Chicago 1, Ill., U.S.A.
J. Appl. Phys.	Journal of Applied Physics, American Institute of Physics. 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Chem. Phys.	The Journal of Chemical Physics, American Institute of Physics, Inc., 57, East 55th Street, New York 22, N.Y., U.S.A.
J. Imp. College Chem. Engng. Soc.	Journal of the Imperial College Chemical Engineering Society. Imperial College Chemical Engineering Society, Editorial Board, Dept. Chemical Engineering, Imperial College, London S.W.7, England.
J. Iron Steel Inst.	Journal of the Iron and Steel Institute, Iron and Steel Institute, 4, Grosvenor Gardens, London S.W.1, England.
J. Med. Lab. Tech.	Journal of Medical Laboratory Technology, Institute of Medical Laboratory Technology, 9, Harley Street, London W.1, England.
J. Metals	Journal of Metals, American Institute of Mining and Metallurgical Engineers, Inc. 29, West 39th Street, New York 18, N.Y., U.S.A.
J. Opt. Soc. Amer.	Journal of the Optical Society of America, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
J Phys. Radium	Journal de Physique et le Radium, La Société Francaise de Physique, 10, rue Vauquelin, Paris 5e, France.
J. Phys. Soc., Japan	Journal of the Physical Society of Japan, Physical Society of Japan, c/o Dept. Physics, Faculty of Science, Tokyo University, Bunkyo-ku, Tokyo, Japan.
J. Res. Nat. Bur. Stand.	Journal of Research of the National Bureau of Standards, U.S. Government Printing Office, Washington 25, D.C., U.S.A.

Vol. 3 1953

Abbreviations and Publishers of Journals Quoted in this Volume
Contd.

J.Sci.Food Agric.	Journal of the Science of Food and Agriculture, Society of Chemical Industry, 56, Victoria Street, London S.W.1, England.
J. Sci. Instrum.	Journal Scientific Instruments, Institute of Physics, 47, Belgrave Square, London S.W.1, England.
Lab. Practice	Laboratory Practice, United Trade Press Ltd., 9, Gough Square, Fleet Street, London E.C.4, England.
Light Metals	Temple Press Ltd., Bowling Green Lane, London E.C.1, England.
Machinist	McGraw-Hill Publishing Co., Ltd., 95, Farringdon Street, London E.C.4, England.
Mater. & Meth.	Materials and Methods, 330, West 42nd Street, New York 18, N.Y., U.S.A.
Mech. World & Engng. Rec.	Mechanical World and Engineering Record, 31, King Street West, Manchester 3, England.
Mém. Soc. Roy. Belge. Industr.	Memoires de la Société Royale Belge des Ingénieurs et des Industriels. Hôtel Ravenstein, 3, rue Ravenstein, Bruxelles, Belgium.
Metal Finish.	Metal Finishing, Finishing Publications, Inc., 11, West 42nd Street, New York 18, N.Y., U.S.A.
Metal Progr.	Metal Progress, The American Society for Metals, 7301, Euclid Avenue, Cleveland 3, Ohio, U.S.A.
Metal Treatm.	Metal Treatment, Industrial Newspapers Ltd., 49, Wellington Street, Strand, London W.C.2, England.
Metall	Metall Verlag Gm.b.H, Hubertusallee 18, Berlin—Grunewald, Germany.
Metallurgia	The Kennedy Press, Ltd., 31, King Street West, Manchester 3, England.
Mill & Factory	205, East 42nd Street, New York 17, N.Y., U.S.A.
Mod. Res. Techniques Phys. Metallurgy	Modern Research Techniques in Physical Metallurgy, Report on Conference held in October, 1952, American Society for Metals, Cleveland, Ohio, U.S.A.
Monatsh.	Monatshefte für Mathematik und Physik, Akademie der Wissenschaften, Vienna, Austria.
Mon. Not. Roy. Astr. Soc.	Monthly Notices of the Royal Astronomical Society, Royal Astronomical Society, Burlington House, London W.1, England.
N.A.C.A. Rep.	National Advisory Committee for Aeronautics, Washington D.C., U.S.A.
Nature	Macmillan and Co., St. Martin's Street, London W.C.2, England.
Naturwiss.	Naturwissenschaften, Springer Verlag, Jebenstrasse 1, Berlin—Charlottenburg, Germany.
N.B.S. Report	National Bureau of Standards Report, Office of Scientific Publications, National Bureau of Standards, Washington 25, D.C., U.S.A.
Nederlands Tijdschr. Natuurk.	Nederlands Tijdschrift voor Natuurkunde, Martinus Nijhoff, Lange Voorhout No. 9, The Hague, Holland.
N. J. Cer. Res. Sta. Rep.	Technical Reports, N.J. Ceramic Research Station, Rutgers University, New Brunswick, N.J., U.S.A.
N.R.C. Canada Rep.	National Research Council of Canada Report, National Research Council of Canada, Ottawa, Canada.
Nuclear Science Abstr.	Nuclear Science Abstracts, U.S. Atomic Energy Commission, Office of Technical Services, Department of Commerce, Washington 25, D.C., U.S.A.
Nucleonics	McGraw-Hill Publishing Co. Inc., 330, West 42nd Street, New York 18, N.Y., U.S.A.
Oak Ridge Nat. Lab. Reports	Oak Ridge National Laboratory Reports, Y-12 Area, Oak Ridge, Tenn., U.S.A.
Optik	Wissenschaftliche Verlagsgesellschaft m.b.H, Stuttgart, Germany.
Paint, Oil, Colour J.	Paint, Oil and Colour Journal, Scott Greenwood & Son Ltd., 83-86, Farringdon Street, London E.C.4, England.
Paper Mkr.	The Paper-Maker and British Paper Trade Journal, S. C. Phillips & Co., Ltd., 3, Tudor Street, London E.C.4, England.
Petrol. Ref.	Petroleum Refiner, The Gulf Publishing Co., Box 2608, Houston 1, Texas, U.S.A.
Pharm. Acta Helv.	Pharmaceutica Acta Helvetiae, Schweitzerischer Apotheker-Verein, Sihil Strasse 37, Zürich, Switzerland.
Philips Tech. Rev.	Philips Technical Review, Philips Electrical Ltd., Century House, Shaftesbury Avenue, London W.C.2, England.

Vol
3
195

Abbreviations and Publishers of Journals Quoted in this Volume
Contd.

Phys. Rev.	Physical Review, American Institute of Physics, Brookhaven National Laboratory, Upton, Long Island, N.Y., U.S.A.
Plansee Proc.	Plansee Proceedings, Metallwerk Plansee G.m.b.H, Reutte/Tyrol, Austria.
Planseeber. Pulvermet.	Planseeberichte für Pulvermetallurgie, Metallwerk Plansee G.m.b.H., Reutte/Tyrol, Austria.
Plastics Progr.	Plastics Progress, Iliffe & Sons Ltd. Dorset House, Stamford Street, London S.E.1, England.
Plating	The American Electroplaters Society, American Building, 445, Broad Street, Newark 2, N.J., U.S.A.
Proc. Amer. Soc. Test. Mater.	Proceedings of the American Society for Testing Materials, The American Society for Testing Materials, 20th and Northampton Streets, Easton, Pa., U.S.A.
Proc. I.E.E.	Proceedings of the Institution of Electrical Engineers, Institution of Electrical Engineers, Savoy Place, London W.C.2, England.
Proc. Phys. Soc.	Proceedings of the Physical Society, The Physical Society, 1, Lowther Gardens, Prince Consort Road, London S.W.7, England.
Proc. Roy. Soc.	Proceedings of the Royal Society, The Royal Society , Burlington House, Piccadilly, London W.1, England.
Prod. Finish.	Product Finishing, Sawell Publication Ltd., 4, Ludgate Circus, London E.C.4, England.
R.C.A. Rev.	R.C.A. Review, R.C.A. Laboratories, Radio Corporation of America, Princeton, N.J., U.S.A.
Refrig. Engng.	Refrigerating Engineering, American Society of Refrigerating Engineers, 40 W. 40th Street, New York 18, N.Y., U.S.A.
Rep. Progr. Phys.	Reports on Progress in Physics, The Physical Society, 1, Lowther Gardens, Prince Consort Road, London S.W.7, England.
Rev. Mod. Phys.	Review of Modern Physics, American Institute of Physics, Lancaster, Pa., U.S.A.
Rev. Prat. Froid	Revue Pratique du Froid, 254, Rue de Vaugirard, Paris 15e., France.
Rev. Sci. Instrum.	Review of Scientific Instruments, American Institute of Physics, Prince and Lemon Streets, Lancaster, Pa., U.S.A.
Science	American Association for the Advancement of Science, 1515, Massachusetts Avenue, N.W., Washington 5, D.C., U.S.A.
Science Abstracts	Institution of Electrical Engineers, Savoy Place, London W.C.2, England.
Sci. Light	Science of Light, Institute for Optical Research, Tokyo University of Education, 4-400, Hyatunin-Cho, Shinjuku-Ku, Tokyo, Japan.
Scient. Amer.	Scientific American, Scientific American, Inc., 2, West 45th Street, New York 36, N.Y., U.S.A.
S.P.E.J.	Society of Plastics Engineers Journal, Society of Plastics Engineers, Suite 116-118, 34 East Putnam Avenue, Greenwich, Conn., U.S.A.
Sprechsaal	Sprechsaal für Keramik Glas Email, Verlag Mueller and Schmidt, Coburg, Germany.
Structural Engr.	The Structural Engineer, Institution of Structural Engineers, 11, Upper Belgrave Street, London S.W.1, England.
Telef. Ztg.	Telefunken Zeitung, Technisch-Wissenschaftliche Mitteilungen, Telefunken Ges. für Drahtlose Telegraphie, 32-34, Nehringdamm, Berlin S.W.61, Germany.
Textile Industr.	Textile Industries, A.W.R.C. Smith, 806, Peachtree Street, N.E., Atlanta, Ga., U.S.A.
The Times (Industr. Suppl.)	'The Times' Industrial Supplement, 'The Times' Publishing Co. Ltd., Printing House Square, London E.C.4, England.
Trans. Faraday Soc.	Transactions of the Faraday Society, The Faraday Society, Aberdeen University Press Ltd., 6, Upper Kirkgate, Aberdeen, Scotland.
Trans. I. Chem. E.	Transactions of the Institution of Chemical Engineers, The Institution of Chemical Engineers, 56, Victoria Street, London S.W.1, England.
Univ. Calif. Rad. Lab. Rep.	University of California Radiation Laboratory Reports, University of California, Berkeley, California, U.S.A.
Le Vide	Le Vide, Société Française des Ingénieurs Techniciens du Vide, 44, Rue de Rennes, Paris 6e., France.

Vol. 3 1953

Abbreviations and Publishers of Journals Quoted in this Volume

Contd.

Weld. J.	Welding Journal, The American Welding Society, 33, West 39th Street, New York 18, N.Y., U.S.A.
World Sci. Rev.	World Science Review, Kenneth de Courcy, Alderbourne Manor, Gerrards Cross, Bucks., England.
Yale J. Biol. Med.	Yale Journal of Biology and Medicine, 333, Cedar Street, New Haven 11, Conn., U.S.A.
Zacchia	Rivista di Medicina Legale e delle Assicurazioni, Viale Dell 'Università, Roma, Italy.
Z. Angew Phys.	Zeitschrift für Angewandte Physik, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.
Z. Phys.	Zeitschrift für Physik, Springer Verlag, Jebenstrasse 1, Berlin-Charlottenburg, Germany.
Z. V.D. I.	Zeitschrift des Vereines Deutscher Ingenieure, Prinz Georg Strasse 77/79 Düsseldorf, Germany.

Vol
3
195